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FILE COVERS 1907 - 5 Oct 2007 VOL 147 ISS 16 FILE LAST UPDATED: 4 Oct 2007 (20071004/ED)

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This file contains CAS Registry Numbers for easy and accurate substance identification.

=> d que 151 L4 1580012 SEA FILE=REGISTRY ABB=ON PLU=ON P/ELS L22 STR

VAR G1=3/6/9/15/12/24/28/33/37

NODE ATTRIBUTES: NSPEC IS RC AT4 IS RC 7 NSPEC ATIS RC ΑT NSPEC NSPEC IS RC ΑT 10 IS RC NSPEC AT13 NSPEC IS RC AT 16 NSPEC IS RC ΑT 18 19 NSPEC IS RC ATNSPEC IS RC AΤ 21 NSPEC IS RC AΤ 22 NSPEC IS RC AT25 NSPEC IS RC AΤ 26 NSPEC IS RC ΑT 27

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NSPEC
        IS RC
                      29
NSPEC
        IS RC
                  AT
                      30
NSPEC
        IS RC
                  AT
                      31
NSPEC
        IS RC
                  AT
                       35
        IS RC
                  AT
                       36
NSPEC
NSPEC
        IS RC
                  ΑT
                       38
CONNECT IS E3
               RC AT
                       3
CONNECT IS E3
               RC AT
                        6
CONNECT IS E3
               RC AT
                       9
CONNECT IS E2
               RC AT
                       12
               RC AT
                       15
CONNECT IS E1
CONNECT IS E3
               RC AT
CONNECT IS E3 RC AT
                      28
CONNECT IS E3 RC AT
                       33
CONNECT IS E3 RC AT
DEFAULT MLEVEL IS ATOM
DEFAULT ECLEVEL IS LIMITED
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GRAPH ATTRIBUTES:

RING(S) ARE ISOLATED OR EMBEDDED NUMBER OF NODES IS 39

STEREO ATTRIBUTES: NONE

L24	6867	SEA FILE=REGISTRY SUB=L4 SSS FUL L22
L25	2789	SEA FILE=CAPLUS ABB=ON PLU=ON L24(L)PREP+NT/RL
L27	378500	SEA FILE=HCAPLUS ABB=ON PLU=ON ACIDS+PFT,NT1/CT
L38	22956	SEA FILE=HCAPLUS ABB=ON PLU=ON BASES+PFT,NT/CT
L40	9	SEA FILE=HCAPLUS ABB=ON PLU=ON L25 AND L38
L44	5873	SEA FILE=HCAPLUS ABB=ON PLU=ON IONIC LIQUIDS+PFT,NT/CT
L47	5	SEA FILE=HCAPLUS ABB=ON PLU=ON L25 AND (L44 OR IONIC(2A)(LIQU
		ID OR FLUID) OR (LIQUID OR MOLTEN) (2A) SALT)
L49	13	SEA FILE=HCAPLUS ABB=ON PLU=ON L47 OR L40
L50	14	SEA FILE=HCAPLUS ABB=ON PLU=ON L25 AND (L27 OR ACID) AND
		(L38 OR BASE) AND SALT
L51	25	SEA FILE=HCAPLUS ABB=ON PLU=ON L49 OR L50

=> d 151 ibib abs hitind hitstr tot

L51 ANSWER 1 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN ACCESSION NUMBER: 2007:619842 HCAPLUS Full-text

DOCUMENT NUMBER: 147:72880

TITLE: Preparation of phosphonium cation containing P-N bond

for ionic liquid

INVENTOR(S): Muraishi, Kazuki; Sueto, Kumiko; Gao, Yuan

PATENT ASSIGNEE(S): Kanto Denka Kogyo Co., Ltd., Japan

SOURCE: PCT Int. Appl., 109pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PAT	KIN	D	DATE	TE APPLICATION NO.							DATE							
					÷	-		·										
WO	2007	0639		A1		2007	0607	1	NO 2	006-	20061130							
	W:	ΑE,	AG,	AL,	AM,	AT,	AU,	ΑZ,	BA,	BB,	BG,	BR,	BW,	BY,	ΒZ,	CA,	CH,	
		CN,	co,	CR,	CU,	CZ,	DE,	DK,	DM,	DZ,	EC,	EE,	EG,	ES,	FI,	GB,	GD,	
		GE,	GH,	GM,	GT,	HN,	HR,	HU,	ID,	IL,	IN,	IS,	JP,	KE,	KG,	KM,	KN,	
	•	KP,	KR,	KZ,	LA,	LC,	LK,	LR,	LS,	LT,	LU,	LV,	LY,	MA,	MD,	MG,	MK,	

```
MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO,
             RS, RU, SC, SD, SE, SG, SK, SL, SM, SV, SY, TJ, TM, TN, TR, TT,
             TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW
         RW: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE,
             IS, IT, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ,
             CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG, BW, GH,
             GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY,
             KG, KZ, MD, RU, TJ, TM
                                            JP 2005-349163
PRIORITY APPLN. INFO.:
                                                                A 20051202
                                            JP 2006-188910
                                                                A 20060710
OTHER SOURCE(S):
                        MARPAT 147:72880
GI
```

- * STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY AVAILABLE VIA OFFLINE PRINT *
- AB Title compds. I [R1-R11 = H, alkyl, alkenyl, etc.; X1-X3 = N, O, S, etc.; with the proviso that two of X1-X3 can not be N simultaneously] were prepared For example, reaction of methylbis(diethylamino)phosphine, e.g., prepared from phosphorous trichloride in 2 steps, with dibutylsulfate followed by treatment with lithium N,N-bis(trifluoromethanesulfonyl)imide afforded compound II, which showed the conductivity of 0.088 Sm-1 at 25°. Compds. I are claimed useful for elec. storage devices, lithium secondary batteries, etc.
- CC 29-7 (Organometallic and Organometalloidal Compounds) Section cross-reference(s): 37, 38, 52, 76
- ST phosphonium cation phosphorous nitrogen bond ionic liq; elec storage device phosphonium cation ionic liq; lithium secondary battery phosphonium cation ionic liq
- IT Capacitors

(double layer; preparation of phosphonium cation containing P-N bond for ionic liquid)

IT Solar cells

(dye-sensitized; preparation of phosphonium cation containing P-N bond for ionic liquid)

IT Secondary batteries

(lithium; preparation of phosphonium cation containing P-N bond for ionic liquid)

IT Actuators

Electrodeposition

Fuel cells

Ionic liquids

Lubricants

Plasticizers

Primary batteries

Sensors

Solvents

(preparation of phosphonium cation containing P-N bond for ionic liquid)

IT Polymers, uses

RL: TEM (Technical or engineered material use); USES (Uses) (preparation of phosphonium cation containing P-N bond for ionic liquid)

74-88-4, reactions ΙT 74-96-4 75-03-6 75-16-1 77-78-1, Dimethyl 78-79-5, reactions 107-08-4 109-89-7, Diethylamine, sulfate 110-68-9 110-70-3 111-33-1 542-69-8 624-78-2 reactions 625-22-9, Dibutyl sulfate 628-17-1 917-54-4 2344-80-1 6482-24-2, 2-Methoxyethyl bromide 7719-12-2, Phosphorous trichloride RL: RCT (Reactant); RACT (Reactant or reagent)

(preparation of phosphonium cation containing P-N bond for ionic liquid) ΙT 685-83-6P 685-93-8P 1069-08-5P 4534-85-4P 6069-36-9P 32294-62-5P 40201-85-2P 40422-29-5P 77785-55-8P 87920-32-9P 79107-36**-**1P 81175-49-7P 83978-38-5P 83978-39-6P 940301-94-0P 940301-97-3P 777943-34-7P 940301-93-9P 940301-98-4P 940302-00-1P 940302-06-7P 940302-07-8P 940302-08-9P 940302-09-0P 940302-10-3P 940302-11-4P 940302-12-5P 940302-13-6P 940302-14-7P 940302-15-8P 940302-16-9P 940302-17-0P 940302-18-1P 940302-19-2P 940302-20-5P 940302-21-6P RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent) (preparation of phosphonium cation containing P-N bond for ionic liquid) ΙT 7664-41-7, Ammonia, reactions 10025-87-3, Phosphoric trichloride RL: RGT (Reagent); RACT (Reactant or reagent) (preparation of phosphonium cation containing P-N bond for ionic liquid) 940301-48-4P 940301-51-9P IT. 940301-50-8P 940301-53-1P 940301-55-3P 940301-57-5P 940301-59-7P 940301-60-0P 940301-61-1P 940301-62-2P 940301-63-3P 940301-64-4P 940301-66-6P 940301-68-8P 940301-70-2P 940301-72-4P 940301-74-6P 940301-76-8P 940301-78-0P 940301-80-4P 940301-84-8P 940301-85-9P 940301-82-6P 940301-83-7P 940301-87-1P 940301-91-7P 940301-92-8P 940301-95-1P 940301-89-3P 940301-96-2P 940302-04-5P 940302-05-6P 940911-61-5P 940302-02-3P 940911-62-6P 940913-64-4P RL: SPN (Synthetic preparation); TEM (Technical or engineered material use); PREP (Preparation); USES (Uses) (preparation of phosphonium cation containing P-N bond for ionic liquid) IT 685-83-6P 685-93-8P 1069-08-5P RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent) (preparation of phosphonium cation containing P-N bond for ionic liquid)

CN

685-83-6 HCAPLUS

Phosphorodiamidous chloride, N,N,N',N'-tetraethyl- (CA INDEX NAME)

Cl Et2N-P-NEt2

RN

RN 685-93-8 HCAPLUS

CN Phosphinous amide, N,N-diethyl-P,P-dimethyl- (7CI, 8CI, 9CI) (CA INDEX NAME)

CN Phosphoramidous dichloride, N, N-diethyl- (CA INDEX NAME)

REFERENCE COUNT: 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L51 ANSWER 2 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN ACCESSION NUMBER: 2006:1176514 HCAPLUS Full-text

DOCUMENT NUMBER:

145:489389

TITLE:

Process for preparation of phosphonium ionic compounds

as ionic liquids

INVENTOR(S):
PATENT ASSIGNEE(S):

Sueto, Kumiko; Omae, Osamu; Gao, Yuan Kanto Denka Kogyo Co., Ltd., Japan

SOURCE:

PCT Int. Appl., 48pp.

CODEN. DIVVD

CODEN: PIXXD2

DOCUMENT TYPE:

Patent

LANGUAGE:

Japanese

FAMILY ACC. NUM. COUNT: 2

PATENT INFORMATION:

PAT	ENT I	NO.			KIN	IND DATE APPLICATI						ION 1	NO.		DATE			
WO	0 2006118232				A1 20061109					WO 2	006-		2	0060	428			
	W:	ΑE,	AG,	AL,	AM,	ΑT,	AU,	ΑZ,	BA,	BB,	BG,	BR,	BW,	BY,	ΒZ,	CA,	CH,	
		CN,	CO,	CR,	CU,	CZ,	DE,	DK,	DM,	DZ,	EC,	EE,	EG,	ES,	FI,	GB,	GD,	
		GE,	GH,	GM,	HR,	HU,	ID,	IL,	IN,	IS,	JP,	KE,	KG,	KM,	KN,	KP,	KR,	
		KZ,	LC,	LK,	LR,	LS,	LT,	LU,	LV,	LY,	MA,	MD,	MG,	MK,	MN,	MW,	MX,	
		MZ,	NA,	NG,	NI,	NO,	NZ,	OM,	PG,	PH,	PL,	PT,	RO,	RU,	SC,	SD,	SE,	
		SG,	SK,	SL,	SM,	SY,	TJ,	TM,	TN,	TR,	TT,	TZ,	UA,	UG,	US,	UZ,	VC,	
		VN,	YU,	ZA,	ZM,	ZW								•				
	RW:	AT,	BE,	BG,	CH,	CY,	CZ,	DE,	DK,	EE,	ES,	FI,	FR,	GB,	GR,	HU,	ΙE,	
		IS,	IT,	LT,	LU,	LV,	MC,	NL,	PL,	PT,	RO,	SE,	SI,	SK,	TR,	BF,	ВJ,	
		CF,	CG,	CI,	CM,	GA,	GN,	GQ,	GW,	ML,	MR,	ΝE,	SN,	TD,	TG,	BW,	GH,	
		GM,	ΚE,	LS,	MW,	MZ,	NA,	.SD,	SL,	SZ,	TZ,	UG,	ZM,	ZW,	AM,	ΑZ,	BY,	
		KG,	KZ,	MD,	RU,	ТJ,	MT											
WO	2006	1178	72		A1	:	2006	1109	1	WO 2	005-	JP82	29		2	0050	428	
	W:	ΑE,	AG,	AL,	AM,	ΑT,	ΑU,	AZ,	BA,	BB,	BG,	BR,	BW,	BY,	BZ,	CA,	CH,	
		CN,	co,	CR,	CU,	CZ,	DE,	DK,	DM,	DZ,	EC,	EE,	EG,	ES,	FI,	GB,	GD,	
		GE,	GH,	GM,	HR,	HU,	ID,	IL,	IN,	IS,	JP,	ΚE,	KG,	KM,	KP,	KR,	KZ,	
		LC,	LK,	LR,	LS,	LT,	LU,	LV,	MA,	MD,	MG,	MK,	MN,	MW,	MX,	MZ,	NA,	
		NI,	NO,	NZ,	OM,	PG,	PH,	PL,	PT,	RO,	RU,	SC,	SD,	SE,	SG,	SK,	SL,	
		SM,	SY,	ТJ,	TM,	TN,	TR,	TT,	TZ,	UA,	ŪG,	US,	UZ,	VC,	VN,	YU,	ZA,	
		ZM,	zw															
	RW:	AT,	BE,	BG,	CH,	CY,	CZ,	DE,	DK,	EE,	ES,	FI,	FR,	GB,	GR,	HU,	IE,	
		IS,	IT,	LT,	LU,	MC,	NL,	PL,	PT,	RO,	SE,	SI,	SK,	TR,	BF,	BJ,	CF,	
		CG,	CI,	CM,	GΑ,	GN,	GQ,	GW,	ML,	MR,	ΝE,	SN,	TD,	TG,	BW,	GH,	GM,	
		KE,	LS,	MW,	MZ,	NA,	SD,	SL,	SZ,	TZ,	UG,	ZM,	ZW,	AM,	ΑZ,	BY,	KG,	
		ΚZ,	MD,	RU,	TJ,	TM												
IORIT	WO 2005-JP8229 A 20050428 MARPAT 145:489389											428						
		, 5 / •			11111	- 4 1-		-000										

```
This invention pertains to a method for producing phosphonium ionic compds.
AΒ
     with general formula of P+(NR2R3)(NR4R5)(NR6R7)(XR1R8R9)•A- [wherein R1-R9 =
     independently H, alkyl, alkenyl, alkynyl, etc.; X = S, O, or C; A = anion],
     which comprises alkylation and anion exchange. For example, PO(NMe2)3 was
     reacted with Me2SO4, followed by the addition of Li+•(CF3SO2)2N- to give
     P+(OMe)(NMe2)3•(CF3SO2)2N- (70% in two steps). The title compds. are useful
     in elec. storage device, lithium secondary batteries, elec. double layer
     capacitor, solar cells, fuel cells, and as reaction solvents.
     29-7 (Organometallic and Organometalloidal Compounds)
CC
     Section cross-reference(s): 72
     prepn phosphonium phosphoric amide ionic liq elec
ST
     storage device
ΙT
     Electric double layer
        (capacitor; preparation of phosphonium ionic compds. as ionic
        liqs.)
ΙT
     Solar cells
        (dye-sensitization type; preparation of phosphonium ionic compds. as
        ionic ligs.)
IT
     Capacitors
        (elec. double layer; preparation of phosphonium ionic compds. as
        ionic liqs.)
IT
     Secondary batteries
        (lithium; preparation of phosphonium ionic compds. as ionic
        liqs.)
IT . Alkylation
     Anion exchange
     Fuel cells
       Ionic liquids
     Secondary batteries
     Solvents
        (preparation of phosphonium ionic compds. as ionic liqs
        .)
                    914300-33-7P
                                   914300-38-2P
                                                   914300-44-0P
IT
     914300-28-0P
     RL: DEV (Device component use); RCT (Reactant); SPN (Synthetic
     preparation); PREP (Preparation); RACT (Reactant or reagent); USES (Uses)
        (preparation of phosphonium ionic compds. as ionic ligs
        . )
IT
     914291-26-2P
                    914291-27-3P
                                   914291-28-4P
                                                   914300-29-1P
                                                                  914300-30-4P
     914300-31-5P
                    914300-34-8P
                                   914300-35-9P
                                                   914300-36-0P
                                                                  914300-39-3P
     914300-40-6P.
                    914300-41-7P
                                   914300-46-2P
                                                   914403-10-4P
     RL: DEV (Device component use); SPN (Synthetic preparation); PREP
     (Preparation); USES (Uses)
         (preparation of phosphonium ionic compds. as ionic liqs
        .)
IT
     64-67-5, Diethyl sulfate
                                77-78-1, Dimethyl sulfate
                                                             110-68-9,
     Methylbutylamine
                        625-22-9, Dibutyl sulfate
                                                     680-31-9,
     Hexamethylphosphoric triamide, reactions
                                                1608-26-0
                                                             72593-05-6
     90076-65-6, Lithium bis(trifluoromethanesulfonyl)imide
     RL: RCT (Reactant); RACT (Reactant or reagent)
        (preparation of phosphonium ionic compds. as ionic ligs
        .)
     16613-97-1P
                   32755-11-6P
                                 914291-29-5P
                                                 914291-30-8P
IT
     914300-52-0P
                    914403-11-5P
     RL: RCT (Reactant); SPN (Synthetic preparation); PREP
     (Preparation); RACT (Reactant or reagent)
         (preparation of phosphonium ionic compds. as ionic liqs
        .)
IT
     914300-52-0P
     RL: RCT (Reactant); SPN (Synthetic preparation); PREP
```

(Preparation); RACT (Reactant or reagent) (preparation of phosphonium ionic compds. as ionic ligs

914300-52-0 HCAPLUS RN

Phosphorous triamide, N,N',N''-tributyl-N,N',N''-trimethyl- (CA INDEX CN

6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS REFERENÇE COUNT: RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L51 ANSWER 3 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN ACCESSION NUMBER: 2006:722283 HCAPLUS Full-text

DOCUMENT NUMBER:

145:336110

TITLE:

A new and convenient method for the synthesis of

strong non-ionic bases

AUTHOR(S):

Taillefer, Marc; Rahier, Nicolas; Hameau, Aurelien;

Volle, Jean-Noel

CORPORATE SOURCE:

Architectures Moleculaires et Materiaux

Nanostructures, UMR CNRS 5076, Ecole Nationale Superieure de Chimie de Montpellier, Montpellier,

F-34296, Fr.

SOURCE:

Chemical Communications (Cambridge, United Kingdom)

(2006), (30), 3238-3239

CODEN: CHCOFS; ISSN: 1359-7345

PUBLISHER:

Royal Society of Chemistry

DOCUMENT TYPE:

Journal

LANGUAGE:

English

CASREACT 145:336110 OTHER SOURCE(S):

- Various strong nonionic phosphazene bases were obtained by a new, efficient and very simple method involving Ph3P:NLi (2) as precursor. 2 Was generated by double deprotonation of la (Ph3PNH2+Cl-), and revealed a strong reactivity towards chlorodiphenylphosphine. Reaction of 2 with 1 equiv Ph2PCl, followed by chlorination with C2Cl6 and subsequent reaction with an alkylamine or gaseous NH3 gave Ph3P:NPPh2NHR+Cl- (5a-c-H+: a-H+: R = H, 87%; b-H+: R = Bn,76%; c-H+: R = tert-Bu, 90%), precursors of the corresponding bases. Subsequent reaction of 5a with BuLi, Ph2PCl, C2Cl6, alkylamine or gaseous NH3 and NaI gave the linear Ph3P:NPh2P:NPPh2NHR+I- (8a-c-H+: a-H+: R = H, 79%; b-H+: R = Bn, 67%; c-H+: R = tert-Bu, 79%), precursors of the corresponding bases. To obtain branched protonated bases, 2 was reacted with 0.5 equiv Ph2PCl. Following the procedure used for 5a-c and linear 8a-c the authors could thus synthesize the branched salt Ph3P:NPhP(N:PPh3)N(tert-Bu)H+I- (10-H+) in 81% yield. Reaction of 2 with 0.25 equiv PCl5 substituted three Cl atoms; subsequent treatment with benzylamine gave the branched salt Ph3P:NP(N:PPh3)2NBnH+Cl- (12-H+) in 58% isolated yield. Determination of the acid-base equilibrium was performed in DMSO with couples 5c/5c-H+ (DMSOpKa = 18.0 ± 0.5), 8c/8c-H+ (DMSOpKa = 19.8 ± 0.5) and 10/10-H+ (DMSOpKa = 23.6 ± 0.5) 0.5).
- 29-7 (Organometallic and Organometalloidal Compounds) CC
- strong nonionic phosphazene base prepn; aminophosphonium chloride deprotonation lithiation chlorodiphenylphosphine amine; phosphine

```
phosphazene chlorination hexachloroethane
IT
     Acidity
     Amination
        (preparation of strong non-ionic phosphazene bases)
IT
     Phosphazenes
     RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP
     (Preparation); RACT (Reactant or reagent)
        (preparation of strong non-ionic phosphazene bases)
IT
     Bases, preparation
     RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)
        (preparation of strong non-ionic phosphazene bases)
IT
     Amines, reactions
     RL: RCT (Reactant); RACT (Reactant or reagent)
        (primary; preparation of strong non-ionic phosphazene bases)
     47869-10-3P 801189-99-1P 910048-49-6P 910048-50-9P
                                                                 910048-51-0P
IT
                   910048-53-2P
                                   910048-54-3P
     910048-52-1P
     RL: PNU (Preparation, unclassified); PREP (Preparation)
         (preparation of strong non-ionic phosphazene bases)
IT
     24082-36-8P
                   910048-38-3P 910048-39-4P
                                               910048-41-8P
     RL: PNU (Preparation, unclassified); RCT (Reactant); PREP
     (Preparation); RACT (Reactant or reagent)
         (preparation of strong non-ionic phosphazene bases)
ΙT
     910048-42-9P
                    910048-46-3P
                                   910048-48-5P
     RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP
     (Preparation); RACT (Reactant or reagent)
         (preparation of strong non-ionic phosphazene bases)
IT
     75-64-9, tert-Butylamine, reactions 100-46-9, Benzylamine, reactions
     603-35-0, Triphenyl phosphine, reactions 1079-66-9,
     Chlorodiphenylphosphine
     RL: RCT (Reactant); RACT (Reactant or reagent)
         (preparation of strong non-ionic phosphazene bases)
     21612-82-8P, Aminotriphenylphosphonium chloride
IT
     910048-40-7P 910048-43-0P
                                  910048-44-1P
                                                 910048-45-2P
                                                                  910048-47-4P
     RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT
      (Reactant or reagent)
         (preparation of strong non-ionic phosphazene bases)
     24082-36-8P 910048-39-4P
IT
     RL: PNU (Preparation, unclassified); RCT (Reactant); PREP
      (Preparation); RACT (Reactant or reagent)
         (preparation of strong non-ionic phosphazene bases)
RN
     24082-36-8 HCAPLUS
CN
     Phosphinous amide, P,P-diphenyl-N-(triphenylphosphoranylidene)- (8CI, 9CI)
        (CA INDEX NAME)
```

Ph 3 P --- N -- P Ph 2

```
RN 910048-39-4 HCAPLUS
CN Phosphinimidic amide, N'-(diphenylphosphino)-N-
(triphenylphosphoranylidene)- (9CI) (CA INDEX NAME)
```

```
Ph
Ph<sub>2</sub>P-N-PPh<sub>3</sub>
```

REFERENCE COUNT:

THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L51 ANSWER 4 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN ACCESSION NUMBER: 2005:1324716 HCAPLUS Full-text

DOCUMENT NUMBER:

144:232984

TITLE:

Ionic liquids-media for unique

phosphorus chemistry

AUTHOR(S):

Amigues, Eric; Hardacre, Christopher; Keane, Gillian;

Migaud, Marie; O'Neill, Maeve

CORPORATE SOURCE:

QUILL and School of Chemistry, Queens University

Belfast, Belfast, BT9 5AG, UK

SOURCE:

Chemical Communications (Cambridge, United Kingdom)

(2006), (1), 72-74

CODEN: CHCOFS; ISSN: 1359-7345

PUBLISHER:

Royal Society of Chemistry

DOCUMENT TYPE: LANGUAGE:

Journal English

OTHER SOURCE(S):

CASREACT 144:232984

AB Ionic liqs. have been shown to offer hitherto unseen control as both a storage solvent for PCl3 and POCl3 and reaction media for fluorination and mixed anhydride formation under benign conditions.

CC 28-9 (Heterocyclic Compounds (More Than One Hetero Atom))
Section cross-reference(s): 29

storage solvent phosphorus chloride fluorination mixed anhydride prepn; phosphorus trichloride oxychloride storage ionic liq; halogen exchange stability phosphorus trichloride oxychloride storage ionic liq; ionic liq media unique

phosphorus chem

IT Ionic liquids

Solvents

Stability

Substitution reaction, nucleophilic

(applications of ionic liqs. as storage solvents

for phosphorous trichloride and phosphorous oxychloride and study of their applicability as reaction media for fluorination and mixed anhydride formation under benign conditions)

IT Halogenation

(transhalogenation; applications of ionic liqs. as storage solvents for phosphorous trichloride and phosphorous oxychloride and study of their applicability as reaction media for fluorination and mixed anhydride formation under benign conditions)

TT 7719-12-2, Phosphorous trichloride 10025-87-3, Phosphoric trichloride 145022-44-2, 1-Ethyl-3-methylimidazolium triflate 145022-45-3,

1-Ethyl-3-methylimidazolium methanesulfonate 174501-65-6,

1-Butyl-3-methylimidazolium tetrafluoroborate 174899-83-3,

1-Butyl-3-methylimidazolium N, N-bis(trifluoromethylsulfonyl) amide

223437-11-4, N,N-Butylmethylpyrolidinium bis(trifluoromethanesulfonyl)amid e 742079-20-5

RL: NUU (Other use, unclassified); RCT (Reactant); RACT (Reactant or reagent); USES (Uses)

(applications of ionic liqs. as storage solvents

for phosphorous trichloride and phosphorous oxychloride and study of their applicability as reaction media for fluorination and mixed anhydride formation under benign conditions)

TT 7664-38-2P, Phosphoric acid, preparation 13537-32-1P, Phosphorofluoridic acid 13779-41-4P, Phosphorodifluoridic acid 13779-42-5P, Phosphorochloridic acid 13779-49-2P, Phosphorodichloridic acid 14939-33-4P, Phosphonochloridic acid 14939-40-3P, Phosphonic dichloride 876179-29-2P 876179-33-8P 876179-37-2P 876179-44-1P 876179-48-5P 876179-53-2P 876179-57-6P 876179-60-1P

RL: PNU (Preparation, unclassified); PREP (Preparation) (applications of ionic liqs. as storage solvents

for phosphorous trichloride and phosphorous oxychloride and study of their applicability as reaction media for fluorination and mixed anhydride formation under benign conditions)

IT 7783-55-3P, Phosphorous trifluoride

RL: SPN (Synthetic preparation); PREP (Preparation)
 (applications of ionic liqs. as storage solvents
 for phosphorous trichloride and phosphorous oxychloride and study of
 their applicability as reaction media for fluorination and mixed
 anhydride formation under benign conditions)

IT 73946-92-6P

RL: SPN (Synthetic preparation); PREP (Preparation) (preparation of tri(imidazolium)phosphine trichloride)

IT 73946-92-6P

RL: SPN (Synthetic preparation); PREP (Preparation) (preparation of tri(imidazolium)phosphine trichloride)

RN 73946-92-6 HCAPLUS

CN 1H-Imidazole, 1,1',1''-phosphinidynetris- (9CI) (CA INDEX NAME)

REFERENCE COUNT: 19 THERE ARE 19 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L51 ANSWER 5 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN ACCESSION NUMBER: 2005:1007246 HCAPLUS Full-text

DOCUMENT NUMBER: 145:145791

TITLE: Selective synthesis of the iminophosphoranes and

phosphorus ylides from (alkylamino)phosphonium salts. Comparative study of electrochemical reduction with

the base method

AUTHOR(S): Okazaki, Yuichi; Takeuchi, Akimasa; Ninomiya,

Yoshihiko; Koketsu, Jungo

CORPORATE SOURCE: Department of Applied Chemistry, College of

Engineering, Chubu University, 1200 Matsumoto-cho,

Kasugai, 487-8501, Japan

SOURCE: Electrochemistry (Tokyo, Japan) (2005), 73(9), 798-806

CODEN: EECTFA; ISSN: 1344-3542

PUBLISHER: Electrochemical Society of Japan

DOCUMENT TYPE: Journal LANGUAGE: English

OTHER SOURCE(S): CASREACT 145:145791

```
AΒ
     Electrochem. reduction of substituted (alkylamino) phosphonium salts was
     carried out to confirm the generations of iminophosphoranes and P ylide, and
     compared with the results of the base method. The Wittig and aza-Wittig
     reaction under the presence of benzaldehyde confirmed the generations of
     iminophosphoranes and P ylides. It is possible to synthesize selectively both
     the iminophosphoranes and the P ylides from a single (alkylamino)phosphonium
     salt by the electrochem. reduction or by the base method under mild
     conditions. As a method of dehydrogenation reaction, the electrochem.
     reduction can play a similar role as strong bases such as Na amide, NaOMe,
     NaOPh, and DBU.
CC
     29-7 (Organometallic and Organometalloidal Compounds)
     Section cross-reference(s): 72
     Bases, reactions
IT
     RL: RGT (Reagent); RACT (Reactant or reagent)
        (effect on chemoselectivity; comparative study of electrochem. reduction
        with base method for selective synthesis of iminophosphoranes and
        phosphorus ylides from (alkylamino)phosphonium salts)
IT
     31036-93-8P, (Isobutylamino)diphenylphosphine 41391-96-2P
     , (Ethylamino)diphenylphosphine 51439-15-7P,
     (Butylamino) diphenylphosphine 382624-25-1P,
     (Cyclohexylamino) diphenylphosphine
     RL: RCT (Reactant); SPN (Synthetic preparation); PREP
     (Preparation); RACT (Reactant or reagent)
        (quaternization; comparative study of electrochem. reduction with base
        method for selective synthesis of iminophosphoranes and phosphorus
        ylides from (alkylamino)phosphonium salts)
     31036-93-8P, (Isobutylamino)diphenylphosphine 41391-96-2P
     , (Ethylamino)diphenylphosphine 51439-15-7,P,
     (Butylamino)diphenylphosphine 382624-25-1P,
     (Cyclohexylamino) diphenylphosphine
     RL: RCT (Reactant); SPN (Synthetic preparation); PREP
     (Preparation); RACT (Reactant or reagent)
        (quaternization; comparative study of electrochem. reduction with base
        method for selective synthesis of iminophosphoranes and phosphorus
        ylides from (alkylamino)phosphonium salts)
RN
     31036-93-8 HCAPLUS
CN
     Phosphinous amide, N-(2-methylpropyl)-P,P-diphenyl- (9CI) (CA INDEX NAME)
 Ph-P-NH-Bu-i
     41391-96-2 HCAPLUS
RN
CN
     Phosphinous amide, N-ethyl-P,P-diphenyl- (7CI, 9CI) (CA INDEX NAME)
    Ph
 Ph-P-NH-Et
```

51439-15-7 HCAPLUS

Phosphinous amide, N-butyl-P, P-diphenyl- (9CI)

RN CN

(CA INDEX NAME)

Ph-P-NH-Bu-n

382624-25-1 HCAPLUS RN

CN Phosphinous amide, N-cyclohexyl-P, P-diphenyl- (9CI) (CA INDEX NAME)

NH-PPh2

REFERENCE COUNT:

THERE ARE 17 CITED REFERENCES AVAILABLE FOR THIS 17 RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L51 ANSWER 6 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:

2004:460850 HCAPLUS Full-text

DOCUMENT NUMBER:

141:277698

TITLE:

Stereoselective Reactions of Chiral Amines with

Racemic Chlorophosphines

AUTHOR(S):

SOURCE:

Gryshkun, Evgenyi V.; Andrushko, Natalia V.;

Kolodiazhnyi, Oleg I.

CORPORATE SOURCE:

National Academy of Sciences of Ukraine, Kiev, Ukraine

Phosphorus, Sulfur and Silicon and the Related

Elements (2004), 179(6), 1027-1046

CODEN: PSSLEC; ISSN: 1042-6507

PUBLISHER:

Taylor & Francis, Inc.

DOCUMENT TYPE:

Journal

LANGUAGE:

English

OTHER SOURCE(S):

CASREACT 141:277698

Racemic chlorophosphines react stereoselectively with chiral 1phenylethylamines or amino acid esters to give diastereomerically enriched aminophosphines 3 (84 %de and 85 % yield for (RP,S)-tBuPhPNHCHMePh from tBuPhPCl and (S)-NH2CHMePh), which were isolated as diastereomerically pure crystalline borane complexes. Oxidation, thionation, the reaction with MeI provide optically active derivs. of aminophosphines. (R,S)- and (S,S)stereoisomers of phosphinic acid amides were separated by crystallization and flash-chromatog. The stereochem. properties of P acid amides were studied. The mechanism of asym. induction at the trivalent P atom was rationalized.

CC 29-7 (Organometallic and Organometalloidal Compounds)

IT Bases, reactions

> RL: RGT (Reagent); RACT (Reactant or reagent) (Bronsted bases, stereoselectivity affected by; stereoselective reactions of chiral amines with racemic chlorophosphines and stereospecific reactions of resulting aminophosphines)

168431-82-1P, Methyl (S)-2-[[(R)-tertbutyl (phenyl) phosphino] amino] -4-methylpentanoate 220812-74-8P,

(S)-P-tert-Butyl-P-phenyl-N-((S)-1-phenylethyl)phosphinous amide

220812-79-3P, (R)-P-tert-Butyl-P-phenyl-N-((S)-1-

phenylethyl)phosphinous amide 538311-43-2P, (R)-P-Mesityl-Pphenyl-N-((S)-1-phenylethyl)phosphinous amide

757955-86-5P 757960-25-1P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP

(Preparation); RACT (Reactant or reagent)

(stereoselective reactions of chiral amines with racemic chlorophosphines and stereospecific reactions of resulting aminophosphines)

168431-86-5P, Methyl (S)-2-[[(R)-tert-butyl(phenyl)phosphinyl]amino]-4-IT methylpentanoate 168431-88-7P, Methyl (S)-2-[[(S)-tertbutyl(phenyl)phosphinyl]amino]-4-methylpentanoate 171776-23-1P, Methyl (S)-2-[[(S)-tert-butyl(phenyl)phosphinothioyl]amino]-4-methylpentanoate 171776-24-2P, Methyl (S)-2-[[(R)-tert-butyl(phenyl)phosphinothioyl]amino]-220812-76-0P, (S)-P-tert-Butyl-P-phenyl-N-((S)-1-4-methylpentanoate phenylethyl)phosphinothioic amide 220812-77-1P, (R)-P-tert-Butyl-Pphenyl-N-((S)-1-phenylethyl)phosphinothioic amide 538311-41-0P, (S)-P-tert-Butyl-P-phenyl-N-((S)-1-phenylethyl)phosphinic amide538311-44-3P, (R)-P-tert-Butyl-P-phenyl-N-((S)-1-phenylethyl)phosphinic amide 757207-98-0P, Methyl (S)-2-[[(R)-tertbutyl(phenyl)phosphino]amino]-3-methylbutanoate 757208-15-4P, (R)-tert-Butyl (methyl) (phenyl) [((S)-1-phenylethyl)amino]phosphonium iodide RL: SPN (Synthetic preparation); PREP (Preparation) (stereoselective reactions of chiral amines with racemic chlorophosphines and stereospecific reactions of resulting aminophosphines)

IT 168431-82-1P, Methyl (S)-2-[[(R)-tertbutyl (phenyl) phosphino] amino] -4-methylpentanoate 220812-74-8P, (S)-P-tert-Butyl-P-phenyl-N-((S)-1-phenylethyl)phosphinous amide 220812-79-3P, (R)-P-tert-Butyl-P-phenyl-N-((S)-1phenylethyl)phosphinous amide 538311-43-2P, (R)-P-Mesityl-Pphenyl-N-((S)-1-phenylethyl)phosphinous amide RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent) (stereoselective reactions of chiral amines with racemic chlorophosphines and stereospecific reactions of resulting

aminophosphines)

RN 168431-82-1 HCAPLUS

CN L-Leucine, N-[(R)-(1,1-dimethylethyl)phenylphosphino]-, methyl ester (9CI) (CA INDEX NAME) .

Absolute stereochemistry.

RN 220812-74-8 HCAPLUS

Phosphinous amide, P-(1,1-dimethylethyl)-P-phenyl-N-[(1S)-1-phenylethyl]-, CN [P(S)]-(9CI) (CA INDEX NAME)

Absolute stereochemistry.

RN220812-79-3 HCAPLUS

Phosphinous amide, P-(1,1-dimethylethyl)-P-phenyl-N-[(1S)-1-phenylethyl]-, CN. [P(R)]- (9CI) (CA INDEX NAME)

Absolute stereochemistry.

RN 538311-43-2 HCAPLUS

CNPhosphinous amide, P-phenyl-N-[(1S)-1-phenylethyl]-P-(2,4,6trimethylphenyl)-, [P(R)]- (9CI) (CA INDEX NAME)

Absolute stereochemistry.

IT 757207-98-0P, Methyl (S)-2-[[(R)-tert-

butyl (phenyl) phosphino] amino] -3-methylbutanoate

RL: SPN (Synthetic preparation); PREP (Preparation)

(stereoselective reactions of chiral amines with racemic

chlorophosphines and stereospecific reactions of resulting

aminophosphines)

757207-98-0 HCAPLUS RN

CN L-Valine, N-[(R)-(1,1-dimethylethyl)phenylphosphino]-, methyl ester INDEX NAME)

Absolute stereochemistry.

REFERENCE COUNT:

33 THERE ARE 33 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L51 ANSWER 7 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN

```
10/500,145
                         2004:194888 HCAPLUS Full-text
ACCESSION NUMBER:
DOCUMENT NUMBER:
                         140:391324
                         Transformation between Diphosphinoamines and
TITLE:
                         Iminobiphosphines: a Reversible P-N-P ↔ N:P-P
                         Rearrangement Triggered by Protonation/Deprotonation
                         Fei, Zhaofu; Biricik, Nermin; Zhao, Dongbin;
AUTHOR(S):
                         Scopelliti, Rosario; Dyson, Paul J.
CORPORATE SOURCE:
                         Institut de Chimie Moleculaire et Biologique, Ecole
                         Polytechnique Federale de Lausanne, EPFL-BCH,
                         Lausanne, CH-1015, Switz.
                         Inorganic Chemistry (2004), 43(7), 2228-2230
SOURCE:
                         CODEN: INOCAJ; ISSN: 0020-1669
                         American Chemical Society
PUBLISHER:
DOCUMENT TYPE:
                         Journal
LANGUAGE:
                         English
                         CASREACT 140:391324
OTHER SOURCE(S):
     Protonation of diphosphinoamines attached to pyridine at the ortho-position
     quant. affords the corresponding iminobiphosphine isomers. For example, 2,6-
      [(Ph2P)2N]2C5H3N reacted with HBF4·Et2O giving 2,6-(Ph2PPh2P:N)2C5H3NH+BF4-.
     The starting material can be recovered quant. by deprotonation with base. The
     system represents a new type of mol. switch. X-ray crystallog. was used to
     establish the structures of 2,6-[Ph2PPh2P:N]2C5H3NH+BF4- and 2-
     (Ph2PPh2P:N) C5H4NH+BF4-.
CC 29-7 (Organometallic and Organometalloidal Compounds)
     Section cross-reference(s): 75
     diphosphinoamine pyridine prepn reversible rearrangement protonation
     tetrafluoroboric trifluoromethanesulfonic acid; iminobiphosphine
     salt prepn structure reversible rearrangement deprotonation
     base; crystal structure iminobiphosphine tetrafluoroborate
     salt; mol structure iminobiphosphine tetrafluoroborate
IT
     Crystal structure
     Molecular structure
        (of iminobiphosphine tetrafluoroborate salts)
     686276-03-9P 686276-04-0P
IT
     RL: RCT (Reactant); SPN (Synthetic preparation); PREP
     (Preparation); RACT (Reactant or reagent)
        (attempted protonation; reversible rearrangement between
```

diphosphinoamines and iminobiphosphines triggered by protonation/deprotonation)

IT 125291-85-2P 644988-94-3P 686276-06-2P 686276-08-4P RL: RCT (Reactant); SPN (Synthetic preparation); PREP

(Preparation); RACT (Reactant or reagent)

(reversible rearrangement between diphosphinoamines and iminobiphosphines triggered by protonation/deprotonation)

686276-03-9P 686276-04-0P IT

RL: RCT (Reactant); SPN (Synthetic preparation); PREP

(Preparation); RACT (Reactant or reagent)

(attempted protonation; reversible rearrangement between diphosphinoamines and iminobiphosphines triggered by protonation/deprotonation)

686276-03-9 HCAPLUS RN

CN Phosphinous amide, N-(diphenylphosphino)-P,P-diphenyl-N-3-pyridinyl- (9CI) (CA INDEX NAME)

RN 686276-04-0 HCAPLUS

CN Phosphinous amide, N-(diphenylphosphino)-P,P-diphenyl-N-4-pyridinyl- (9CI) (CA INDEX NAME)

IT 125291-85-2P 644988-94-3P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP

(Preparation); RACT (Reactant or reagent)

(reversible rearrangement between diphosphinoamines and iminobiphosphines triggered by protonation/deprotonation)

RN 125291-85-2 HCAPLUS

CN Phosphinous amide, N-(diphenylphosphino)-P,P-diphenyl-N-2-pyridinyl- (9CI) (CA INDEX NAME)

RN 644988-94-3 HCAPLUS

CN Phosphinous amide, N,N'-2,6-pyridinediylbis[N-(diphenylphosphino)-P,P-diphenyl- (9CI) (CA INDEX NAME)

REFERENCE COUNT:

33 THERE ARE 33 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L51 ANSWER 8 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN

```
ACCESSION NUMBER:
```

2003:931305 HCAPLUS Full-text

DOCUMENT NUMBER:

140:4839

TITLE:

Process for hydrogenating or asymmetrical

hydrogenating unactivated imines into amines using

ruthenium complexes as catalysts

INVENTOR(S):

Abdur-Rashid, Kamaluddin; Morris, Robert H.

PATENT ASSIGNEE(S):

SOURCE:

PCT Int. Appl., 31 pp.

CODEN: PIXXD2

DOCUMENT TYPE: LANGUAGE:

Patent English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION: DAMENIA NO

	PA'	CENT	NO.			KIN	D	DATE				ICAT:		DATE					
,	WO 2003097571			A1 20031127								20030515							
	W: AE, AG, AL,		AM,	ΑT,	AU,	ΑZ,	BA,	BB,	BG,	BR,	BY,	ΒZ,	CA,	CH,	CN,				
			co,	CR,	CU,	CZ,	DE,	DK,	DM,	DZ,	EC,	EE,	ES,	FI,	GB,	GD,	GE,	GH,	
			GM,	HR,	HU,	ID,	IL,	IN,	IS,	JP,	KE,	KG,	KP,	KR,	ΚZ,	LC,	LK,	LR,	
			LS,	LT,	LU,	LV,	MA,	MD,	MG,	MK,	MN,	MW,	MX,	MZ,	NI,	NO,	NZ,	OM,	
			ΡH,	PL,	PT,	RO,	RU,	SC,	SD,	SE,	SG,	SK,	SL,	TJ,	TM,	TN,	TR,	TT,	
			TZ,	UA,	UG,	US,	UZ,	VC,	VN,	YU,	ZA,	ZM,	ZW						
		RW:	GH,	GM,	KE,	LS,	MW,	MZ,	SD,	SL,	SŻ,	TZ,	UG,	ZM,	ZW,	AM,	ΑZ,	BY,	
			KG,	ΚŻ,	MD,	RU,	ТJ,	TM,	AT,	BE,	BG,	CH,	CY,	CZ,	DE,	DK,	EE,	ES,	
			FI,	FR,	GB,	GR,	HU,	IE,	IT,	LU,	MC,	NL,	PT,	RO,	SE,	SI,	SK,	TR,	
			BF,	ВJ,	CF,	CG,	CI,	CM,	GΑ,	GN,	GQ,	GW,	ML,	MR,	NE,	SN,	TD,	TG	
	CA	2489	158			A1		2003	1127		CA 2	20030515							
	AU	2003	2238	06		· A1		2003	1202	:	AU 2	003-	2238	20030515					
	EΡ	1503	979			A 1		2005	0209		EP 2	003-	7200	57	20030515				
		R:	ΑT,	BE,	CH,	DE,	DK,	ES,	FR,	GB,	GR,	IT,	LI,	LU,	NL,	SE,	MC,	PT,	
			ΙE,	SI,	LT,	LV,	FI,	RO,	MK,	CY,	AL,	TR,	BG,	CZ,	EE,	HU,	SK		
	JP	2005	5254	26		T		2005	0825		JP 2	004-	5053	05		2	0030	515	
	US 2005209487					A1	A1 20050922				US 2	005-	5133	21	20050601				
	US	7256	311			B2		2007	0814										
PRIOR	IT:	Y APP	LN.	INFO	.:					,	US 2002-380256P			56P]	P 20020515			
•	·									1	WO 2	003-	CA68	9	1	W 20030515			
OMITTE		STIDAR	101 -			070		m 14	0 . 40	20.		200 1	40 4	0 2 0					

OTHER SOURCE(S): CASREACT 140:4839; MARPAT 140:4839

- A process is described for the hydrogenation or asym. hydrogenation of dialkyl-, alkylalkenyl-, and dialkenyl-imines [e.g., N-(1,2,2trimethylpropylidene)aniline] into the corresponding amines using a catalytic system comprising a base (e.g., potassium isopropoxide) and a ruthenium complex containing (1) a diamine and (2) a diphosphine ligand or monodentate phosphine ligands [e.g., RuHCl(R-BINAP)(R,R-DPEN)] in hydrogenation and asym. hydrogenation processes.
- IC ICM C07C209-52 ICS C07C211-48
- 25-4 (Benzene, Its Derivatives, and Condensed Benzenoid Compounds) Section cross-reference(s): 45, 67
- IT Bases, reactions

RL: RGT (Reagent); RACT (Reactant or reagent)

(process for hydrogenating or asym. hydrogenating unactivated imines into amines using ruthenium complexes as catalysts prepared from)

ΙT 627502-59-4P 628729-41-9P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP

(Preparation); RACT (Reactant or reagent)

(process for hydrogenating or asym. hydrogenating unactivated imines into amines using ruthenium complexes as catalysts prepared from)

627502-59-4P IT

RL: RCT (Reactant); SPN (Synthetic preparation); PREP

(Preparation); RACT (Reactant or reagent)

(process for hydrogenating or asym. hydrogenating unactivated imines into amines using ruthenium complexes as catalysts prepared from)

RN 627502-59-4 HCAPLUS

CN Phosphinous amide, N,N'-(1R,2R)-1,2-cyclohexanediylbis[P,P-dicyclohexyl-(9CI) (CA INDEX NAME)

Absolute stereochemistry.

REFERENCE COUNT:

2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L51 ANSWER 9 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:

2003:591192 HCAPLUS Full-text

DOCUMENT NUMBER:

139:149757

TITLE:

Method for the separation of acids from

chemical reaction mixtures by means of ionic

fluids

INVENTOR(S):

Volland, Martin; Seitz, Verena; Maase, Matthias; Flores, Miguel; Papp, Rainer; Massonne, Klemens;

Stegmann, Veit; Halbritter, Klaus; Noe, Ralf; Bartsch,

Michael; Siegel, Wolfgang; Becker, Michael;

Huttenloch, Oliver

PATENT ASSIGNEE(S):

Basf Aktiengesellschaft, Germany

SOURCE:

PCT Int. Appl., 111 pp.

CODEN: PIXXD2

DOCUMENT TYPE:

Patent

LANGUAGE:

German .

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PAT	ENT :	NO.			KIN	D :	DATE			APPL	ICAT:	DATE					
WO 2003062251 .					A1	-	2003	0731	. 1	WO 2	: 003-:	 EP54	9		2	0030	121
	W:	ΑE,	AG,	AL,	AM,	AT,	AU,	AZ,	BA,	BB,	BG,	BR,	BY,	BZ,	CA,	CH,	CN,
		co,	CR,	CU,	CZ,	DE,	DK,	DM,	DZ,	EC,	EE,	ES,	FI,	GB,	GD,	GE,	GH,
		GM,	HR,	HU,	ID,	IL,	IN,	IS,	JP,	KE,	KG,	KP,	KR,	KZ,	LC,	LK,	LR,
		LS,	LT,	LU,	LV,	MA,	MD,	MG,	MK,	MN,	MW,	MX,	MZ,	NO,	NZ,	OM,	PH,
		PL,	PT,	RO,	RU,	SC,	SD,	SE,	SG,	SK,	SL,	TJ,	TM,	TN,	TR,	TT,	TZ,
		UA,	UG,	US,	UZ,	VC,	VN,	YU,	ZA,	ZM,	zw						•
	RW:	GH,	GM,	KE,	LS,	MW,	MZ,	SD,	SL,	SZ,	TZ,	ŪG,	ZM,	ZW,	AM,	AZ,	BY,
		KG,	KZ,	MD,	RU,	ТJ,	TM,	AT,	BE,	BG,	CH,	CY,	CZ,	DE,	DK,	EE,	ES,
		FI,	FR,	GB,	GR,	HU,	IE,	IT,	LU,	MC,	NL,	PT,	SE,	SI,	SK,	TR,	BF,
		ВJ,	CF,	CG,	CI,	CM,	GA,	GN,	GO,	GW,	ML.	MR.	NE.	SN.	TD.	TG	

OTHER SOURCE(S):

DE 10202838

DE 10230222

DE 10248902

DE 10251140

CA 2473954

EP 1470136

EP 1470136

CN 1622948

AT 358134 -

JP 2005515258

US 2005020857

ZA 2004006664

PRIORITY APPLN. INFO.:

Disclosed is a method for producing aminodihalophosphines, AB diaminohalophosphines, triaminophosphines, phosphite diamides, aminophosphines, diaminophosphines, phosphite amide halogenides, and aminophosphine halogenides by separating an acid in the presence of an auxiliary base. Said auxiliary base (b) forms a salt with an acid, which is liquid at temps. at which the valuable product is not significantly decomposed during separation of the liquid salt, and (c) the salt of the auxiliary base and the valuable product or the solution of the valuable product form two immiscible phases in a suitable solvent. Thus, reaction of dichloro(phenyl)phosphine with EtOH in presence of 1-methylimidazole (auxiliary base) followed by separation of immiscible i-methylimidazole containing ionic liquid gave up to 96% of diethoxyphenylphosphine.

IC ICM C07F009-22 ICS C07B063-00

CC 29-7 (Organometallic and Organometalloidal Compounds) Section cross-reference(s): 21

A1

A1

· A1

A1

Α1

A1

В1

Т

A.

Т

A1

Α

ST acid sepn chem reaction auxiliary base contg ionic fluid; aminodihalophosphine diaminohalophosphine triaminophosphine phosphite amide aminophosphine diaminophosphine prepn; auxiliary base mediated chem reaction

IT Fluids

Organic synthesis

Separation

(method for separation of acids with auxiliary base from chemical reaction mixts. by means of ionic fluids in organic synthesis)

IT Acids, reactions

Bases, reactions

RL: RCT (Reactant); RACT (Reactant or reagent) (method for separation of acids with auxiliary base from chemical reaction mixts. by means of ionic fluids in organic synthesis)

IT 71-36-3, 1-Butanol, reactions 123-75-1, Pyrrolidine, reactions RL: RCT (Reactant); RACT (Reactant or reagent) (acetylation; method for separation of acids with auxiliary base from chemical reaction mixts. by means of ionic

fluids in organic synthesis)

1521-51-3, 3-Bromocyclohexene IT

> RL: RCT (Reactant); RACT (Reactant or reagent) (dehydrobromination; method for separation of acids with auxiliary

```
base from chemical reaction mixts. by means of ionic
        fluids in organic synthesis)
     106-98-9, 1-Butene, reactions
IT
     RL: RCT (Reactant); RACT (Reactant or reagent)
        (hydroformylation; method for separation of acids with auxiliary
        base from chemical reaction mixts. by means of ionic
        fluids in organic synthesis)
IT
     14874-82-9
     RL: CAT (Catalyst use); USES (Uses)
        (method for separation of acids with auxiliary base from
        chemical reaction mixts. by means of ionic fluids in
        organic synthesis)
     64-17-5, Ethanol, reactions
                                   68-26-8, all-trans-Retinol
                                                                75-84-3,
IT
     Neopentyl alcohol 78-83-1, Isobutanol, reactions
                                                         78-92-2, 2-Butanol
     83-34-1, 3-Methylindole 88-18-6, 2-tert-Butylphenol 90-43-7,
     [1,1'-Biphenyl]-2-ol 100-51-6, Benzyl alcohol, reactions
                                                123-54-6, Acetylacetone,
              112-67-4, Hexadecanoyl chloride
     2-Butene
                 462-06-6, Fluorobenzene 556-82-1, Prenol
                                                              644 - 97 - 3,
     reactions
     Dichloro(phenyl)phosphine
                                760-67-8, 2-Ethylhexanoic acid
                931-40-8, 4-(Hydroxymethyl)-1,3-dioxolan-2-one
                                                                 1079-66-9,
                                                                   22277-50-5
     Chlorodiphenylphosphine 7719-12-2, Phosphorus trichloride
                               472986-87-1 571170-98-4
     26567-10-2
                  72102-69-3
                                                           571170-99-5
     571171-01-2
     RL: RCT (Reactant); RACT (Reactant or reagent)
        (method for separation of acids with auxiliary base from
        chemical reaction mixts. by means of ionic fluids in
        organic synthesis)
IT
     571170-97-3P 571171-04-5P
     RL: RCT (Reactant); SPN (Synthetic preparation); PREP
     (Preparation); RACT (Reactant or reagent)
        (method for separation of acids with auxiliary base from
        chemical reaction mixts. by means of ionic fluids in
        organic synthesis)
     100-71-0, 2-Ethylpyridine 102-82-9, Tributylamine
IT
                                                           109 - 0.6 - 8,
     2-Methylpyridine
                       121-44-8, Triethylamine, reactions
                                                             616-47-7,
     1-Methylimidazole 3001-72-7, 1,5-Diazabicyclo[4.3.0]non-5-ene
     4316-42-1, 1-Butylimidazole
                                   6703-22-6
     RL: RGT (Reagent); RACT (Reactant or reagent)
        (method for separation of acids with auxiliary base from
        chemical reaction mixts. by means of ionic fluids in
        organic synthesis)
                                  105-46-4P, 2-Butyl acetate
IT
     78-10-4P, Tetraethoxysilane
                        110-62-3P, Valeraldehyde 122-52-1P, Triethyl
     Isobutyl acetate
                 123-86-4P, Butyl acetate
                                           136-60-7P, Butyl benzoate
     phosphite
                                  592-57-4P, 1,3-Cyclohexadiene
     590-86-3P, Isovaleraldehyde
                                                                   719-80-2P,
                              926-41-0P, Neopentyl acetate
     Ethoxydiphenylphosphine
                                                              1638-86-4P,
     Diethoxy(phenyl)phosphine 1825-65-6P, 1-Trimethylsilyloxybutane
     1825-66-7P, 2-Trimethylsilyloxybutane 4030-18-6P, N-Acetylpyrrolidine
     13257-81-3P, 4-Trimethylsilyloxypent-3-en-2-one
                                                     14642-79-6P, Benzyl
                            18246-63-4P
                                          35487-17-3P
                                                        78405-71-7P
     trimethylsilyl ether
     86178-32-7P 91993-35-0P, Dichloro(fluorophenyl)phosphine
                                                                  188667-38-1P
                    220472-84-4P 472986-82-6P
                                               509083-87-8P
     205490-65-9P
                                  528597-72-0P
                                                  571171-00-1P
                                                                 571171-02-3P
     509095-18-5P
                    512172-95-1P
     571171-03-4P
     RL: SPN (Synthetic preparation); PREP (Preparation)
        (method for separation of acids with auxiliary base from
        chemical reaction mixts. by means of ionic fluids in
        organic synthesis)
     571171-04-5P
```

RL: RCT (Reactant); SPN (Synthetic preparation); PREP

(Preparation); RACT (Reactant or reagent) (method for separation of acids with auxiliary base from chemical reaction mixts. by means of ionic fluids in organic synthesis)

RN571171-04-5 HCAPLUS

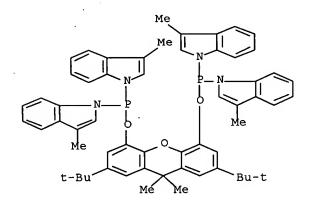
CN Phosphinous chloride, bis(3-methyl-1H-indol-1-yl)- (9CI) (CA INDEX NAME)

472986-82-6P IT

> RL: SPN (Synthetic preparation); PREP (Preparation) (method for separation of acids with auxiliary base from chemical reaction mixts. by means of ionic fluids in organic synthesis)

RN 472986-82-6 HCAPLUS

Phosphinous acid, bis(3-methyl-1H-indol-1-yl)-, 2,7-bis(1,1-dimethylethyl)-CN 9,9-dimethyl-9H-xanthene-4,5-diyl ester (9CI) (CA INDEX NAME)



REFERENCE COUNT: THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L51 ANSWER 10 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:

1999:267972 HCAPLUS Full-text

DOCUMENT NUMBER:

131:19061

TITLE:

Free and Supported Phosphorus Ylides as Strong Neutral

AUTHOR(S):

Bronsted Bases

Goumri-Magnet, Stephanie; Guerret, Olivier; Gornitzka, Heinz; Cazaux, Jean Bernard; Bigg, Dennis; Palacios,

Francisco; Bertrand, Guy

CORPORATE SOURCE:

Laboratoire de Chimie de Coordination, Toulouse,

31077, Fr.

SOURCE:

Journal of Organic Chemistry (1999), 64(10), 3741-3744

CODEN: JOCEAH; ISSN: 0022-3263

PUBLISHER:

American Chemical Society

DOCUMENT TYPE: LANGUAGE: OTHER SOURCE(S): Journal English

CASREACT 131:19061

II

GΙ

$$C1$$
 Ph
 $C1$
 $C1$

To a dimethoxymethane solution of P(NMe2)3 was added at room temperature 2-iodopropane. The solution was stirred under reflux for 72 h, producing [P(NMe2)3Pr-i]I in 91% yield. Potassium hydride was added at 0° to a suspension of [P(NMe2)3Pr-i]I in THF and stirred at room temperature, forming (NMe2)3P:C(Me)2 in 75% yield. A THF solution of (NMe2)3P:C(Me)2 was then added at -78° to a THF solution of benzodiazepines I (R = Me, CH2CO2t-Bu, or CH2Ph) and stirred at room temperature for 1 h. Alkyl halides R'X (R = CH2Ph, CH2CO2t-Bu, or Me), (X = Br or I) were then added and the solution was stirred for an addnl. hour, producing benzodiazepines II (same R' and R) in 38-67% yield. An x-ray crystal structure of II (R = R' = CH2Ph), (space group C222(1), Z = 8, wR2 = 0.3114) was determined The pKa value of [P(NMe2)3Pr-i]I was found to be between 26 and 28 using 31P NMR spectroscopy. The use of ylides as strong nonnucleophilic bases was investigated by reaction of P(NMe2)3 with Merrifield's resin.

CC 29-7 (Organometallic and Organometalloidal Compounds)
Section cross-reference(s): 75

IT Bases, preparation

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(Bronsted bases; preparation and use in benzodiazepine reactions)

IT 1608-26-0DP, reaction products with Merrifield's resin RL: RCT (Reactant); SPN (Synthetic preparation); PREP

(Preparation); RACT (Reactant or reagent)

(reaction with 2-iodopropane)

(reaction with 2-iodopropane)

RN 1608-26-0 HCAPLUS

CN Phosphorous triamide, N,N,N',N',N'',hexamethyl- (CA INDEX NAME)

NMe₂ Me₂N-P-NMe₂

REFERENCE COUNT:

33 THERE ARE 33 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L51 ANSWER 11 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1996:744515 HCAPLUS Full-text

DOCUMENT NUMBER: 126:149660

TITLE:

PUBLISHER:

Room temperature inorganic "quasi-molten

salts" as alkali-metal electrolytes

AUTHOR(S): Xu, K.; Zhang, S.; Angell, C. A.

CORPORATE SOURCE: Dep. Chem., Arizona State Univ., Tempe, AZ,

85287-1604, USA

SOURCE: Journal of the Electrochemical Society (1996),

143(11), 3548-3554

CODEN: JESOAN; ISSN: 0013-4651

Electrochemical Society

DOCUMENT TYPE: Journal LANGUAGE: English

Room temperature inorg. liqs. of high ionic conductivity were prepared by reacting Lewis acid AlCl3 with sulfonyl chlorides. The mechanism is not clear at this time since a crystal structure study of the 1:1 complex with CH3SO2Cl (Tm = 30°) is not consistent with a simple chloride transfer to create AlClO4anions. The liquid is in a state somewhere between ionic and mol. A new term quasi-molten salt is adopted to describe this state. A comparably conducting liquid can be made using BC13 in place of AlC13. Unlike their organic counterparts based on ammonium cations (e.g., pyridinium or imidazolium) which reduce in the presence of alkali metals, this inorg. class of cation shows great stability against electrochem. reduction (.apprx.-1.0 V vs. Li+/Li), with the useful consequence that reversible lithium and sodium metal deposition/stripping can be supported. The electrochem. window for these quasi-salts with AlCl3 ranges up to 5.0 V, and their room temperature conductivities exceed 10-4 S/cm. They dissolve lithium and sodium tetrachloroaluminates up to mole fraction .apprx.0.6 at 100° and intermediate compns. are permanently stable at ambient. The resultant lithium or sodium salt solns. exhibit electrochem. windows of 4.5-5.0 V vs. Li+/Li or Na+/Na and show room temperature conductivities of 10-30 .apprx. 10-25 S/cm. In preliminary charge/discharge tests, the cell Li/quasi- ionic liquid electrolyte/Lil+xMn204 showed a discharge capacity of .apprx.110 mA-h/(g of cathode) and sustained 80% of the initial capacity after 60 cycles, indicating that these quasi- molten salt-based electrolytes are promising candidates for alkali-metal batteries.

CC 72-2 (Electrochemistry)

Section cross-reference(s): 52, 68, 76

ST room temp inorg quasi molten salt; alkali metal electrolyte quasi molten salt; sulfonyl aluminum chloride melt electrochem window; phosphoryl aluminum chloride melt electrochem window; electrochem potential window sulfonyl phosphoryl chloroaluminate; battery electrolyte inorg quasi molten salt

IT Electric potential

(electrochem. potential window of room temperature inorg. quasimolten salts from aluminum chloride and sulfonyl chloride or phosphoryl chloride)

IT 186696-36-6P

RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation)
; PREP (Preparation); RACT (Reactant or reagent)
 (electrochem. potential window and room temperature inorg. quasi-

(electrochem, potential window and room temperature inorg, quasimolten salts as alkali-metal electrolytes)

IT 186696-38-8P 186696-40-2P 186696-41-3P

186696-43-5P

RL: PRP (Properties); SPN (Synthetic preparation); PREP (Preparation)

(ionic conductivity and electrochem. potential window and room temperature inorg. $% \left(1\right) =\left(1\right) \left(1\right)$

10/500,145 quasi-molten salts as alkali-metal electrolytes) IT 75-36-5, Acetyl chloride 124-63-0, Methanesulfonyl chloride RL: PRP (Properties); RCT (Reactant); RACT (Reactant or reagent) (reaction with aluminum chloride: electrochem. potential window and room temperature inorg. quasi-molten salts as alkali-metal electrolytes) IT 6041-61-8P 13966-08-0P 14700-21-1P, Trichlorophosphazosulfonyl chloride RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent) (reaction with aluminum chloride: electrochem. potential window and room temperature inorg. quasi-molten salts as alkali-metal electrolytes) IT 7446-70-0, Aluminum chloride, reactions RL: RCT (Reactant); RACT (Reactant or reagent) (reaction with sulfonyl chloride or phosphoryl chloride for quasimolten salts) IT 186696-36-6P RL: PRP (Properties); RCT (Reactant); SPN (Synthetic preparation) ; PREP (Preparation); RACT (Reactant or reagent) (electrochem. potential window and room temperature inorg. quasimolten salts as alkali-metal electrolytes) RN 186696-36-6 HCAPLUS ÇN Phosphorus(1+), dichloro[ethanaminato(2-)]-, tetrachloroborate(1-) (9CI) (CA INDEX NAME) CM 1 CRN 186696-35-5 CMF C2 H5 C12 N P C12+P == N-Et CM 2 CRN 14911-67-2 CMF B C14 CCI CCS

186696-38-8P 186696-40-2P 186696-43-5P RL: PRP (Properties); SPN (Synthetic preparation); PREP (ionic conductivity and electrochem. potential window and room temperature inorg. quasi-molten salts as alkali-metal electrolytes)

RN 186696-38-8 HCAPLUS

CN Phosphorus(1+), dichloro[methanaminato(2-)]-, (T-4)-tetrachloroaluminate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 186696-37-7 CMF C H3 Cl2 N P

CM 2

CRN 17611-22-2 CMF Al Cl4 CCI CCS

RN 186696-40-2 HCAPLUS

CM 1

CRN 186696-39-9 CMF Cl3 N O2 P S

CM 2

CRN 17611-22-2 CMF Al Cl4 CCI CCS

RN 186696-43-5 HCAPLUS

CN Phosphorus(1+), dichloro[phosphoramidic dichloridato(2-)- κ N]-, (T-4)-tetrachloroaluminate(1-) (9CI) (CA INDEX NAME)

CM 1

CRN 186696-42-4 CMF Cl4 N O P2

CM 2

CRN 17611-22-2 CMF Al C14 CCI CCS

L51 ANSWER 12 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:

1993:39066 HCAPLUS Full-text

DOCUMENT NUMBER:

118:39066

TITLE:

Protonated aminophosphines

AUTHOR(S):

Nifant'ev, E. E.; Gratchev, M. K.; Burmistrov, S. Yu.;

Antipin, M. Yu.; Struchkov, Yu. T.

CORPORATE SOURCE:

V. I. Lenin Pedagog. State Univ., Moscow, 119882,

Russia

SOURCE:

Phosphorus, Sulfur and Silicon and the Related

Elements (1992), 70(1-2), 159-74

CODEN: PSSLEC; ISSN: 1042-6507

DOCUMENT TYPE:

Journal

LANGUAGE:

English

OTHER SOURCE(S):

CASREACT 118:39066

Reaction of tetrafluoroboric acid with aminophosphines R2PNR12 (R = NEt2, R1 = Et; R = NR12 = piperidino; R = Me2CH, Me3C, R1 = Et; R = Me3C, R1 = pyrrol-1-

CC

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RN

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yl) in Et2O give aminophosphonium salts R2P+H(NR12) BF4- in 67-93% yield. NMR
spectroscopy and x-ray anal. of some reactants and products demonstrate that
the protonation occurs at the phosphorus atom only. All aminophosphonium
salts prepared appear not to phosphorylate nucleophiles, whereas
phosphorylation occurs with added base. Thus, reaction of (Et2N) 3P+H BF4-
with PhCHO in CH2Cl2 in the presence of Et3N gave (Et2N)2P(O)CH(NEt2)Ph and
(Et2N) 2P(O) H.
29-7 (Organometallic and Organometalloidal Compounds)
Section cross-reference(s): 75
Protonation and Proton transfer reaction
   (of aminophosphines with tetrafluoroboric acid or pyridinium
   tetrafluoroborate)
139190-39-9, Di-tert-butyl(pyrrol-1-yl)phosphine
RL: PROC (Process)
   (crystal structure and protonation of, with tetrafluoroboric
   acid)
100-52-7, Benzaldehyde, reactions
RL: RCT (Reactant); RACT (Reactant or reagent)
   (phosphorylation of, with aminophosphonium salt)
139190-41-3P 139190-42-4P
RL: PRP (Properties); SPN (Synthetic preparation); PREP
(Preparation)
   (preparation and crystal structure of)
36050-94-9P 126201-43-2P 139190-40-2P
RL: SPN (Synthetic preparation); PREP (Preparation)
   (preparation of)
121-45-9P, Trimethyl phosphite
RL: SPN (Synthetic preparation); PREP (Preparation)
   (preparation of, from methanolysis of aminophosphonium salt)
90532-83-5P
RL: SPN (Synthetic preparation); PREP (Preparation)
   (preparation of, in reaction of aminophosphonium salt with
   benzaldehyde)
139190-38-8P
RL: SPN (Synthetic preparation); PREP (Preparation)
   (preparation, crystal structure, and protonation of, with tetrafluoroboric
   acid)
126450-23-5P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP
(Preparation); RACT (Reactant or reagent)
   (preparation, methanolysis, and phosphorylation with, of benzaldehyde)
12408-02-5
RL: RCT (Reactant); RACT (Reactant or reagent)
   (protonation and Proton transfer reaction, of aminophosphines with
   tetrafluoroboric acid or pyridinium tetrafluoroborate)
2283-11-6, Hexaethylphosphorous triamide
                                           13954-38-6
                                                        65768-04-9
RL: RCT (Reactant); RACT (Reactant or reagent)
   (protonation of, with tetrafluoroboric acid)
505-07-7, Pyridinium tetrafluoroborate 16872-11-0, Tetrafluoroboric
RL: RCT (Reactant); RACT (Reactant or reagent)
   (protonation with, of aminophosphine)
139190-41-3P 139190-42-4P
RL: PRP (Properties); SPN (Synthetic preparation); PREP
(Preparation)
   (preparation and crystal structure of)
139190-41-3 HCAPLUS
```

Phosphinous amide, P, P-bis(1,1-dimethylethyl)-N, N-diethyl-,

mono[tetrafluoroborate(1-)] (9CI) (CA INDEX NAME)

CM 1

CRN 139190-38-8 CMF C12 H28 N P

CM 2

CRN 16872-11-0 CMF B F4 . H CCI CCS

● H+

RN 139190-42-4 HCAPLUS

CN 1H-Pyrrole, 1-[bis(1,1-dimethylethyl)phosphino]-, mono[tetrafluoroborate(1-)] (9CI) (CA INDEX NAME)

CM 1

CRN 139190-39-9 CMF C12 H22 N P

CM 2

CRN 16872-11-0 CMF B F4 . H CCI CCS

H+

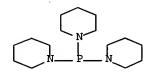
CCI

CCS

● H+

CM 2

CRN 13954-38-6 CMF C15 H30 N3 P



RN 139190-40-2 HCAPLUS
CN Phosphinous amide, N,N-diethyl-P,P-bis(1-methylethyl)-,
mono[tetrafluoroborate(1-)] (9CI) (CA INDEX NAME)

CM 1

CRN 65768-04-9

CMF C10 H24 N P

CM 2

CRN 16872-11-0 CMF B F4 . H CCI CCS

● H+

IT 139190-38-8P

RL: SPN (Synthetic preparation); PREP (Preparation) (preparation, crystal structure, and protonation of, with tetrafluoroboric acid)

RN 139190-38-8 HCAPLUS

CN Phosphinous amide, P,P-bis(1,1-dimethylethyl)-N,N-diethyl- (9CI) (CA INDEX NAME)

IT 126450-23-5P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP

(Preparation); RACT (Reactant or reagent)

(preparation, methanolysis, and phosphorylation with, of benzaldehyde)

RN 126450-23-5 HCAPLUS

CN Phosphorous triamide, hexaethyl-, mono[tetrafluoroborate(1-)] (9CI) (CA INDEX NAME)

CM 1

CRN 16872-11-0 CMF B F4 . H CCI CCS

● H+

CM 2

CRN 2283-11-6 CMF C12 H30 N3 P

NEt2 Et2N-P-NEt2

L51 ANSWER 13 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:

1990:7649 HCAPLUS Full-text

DOCUMENT NUMBER:

112:7649

TITLE:

Synthesis, structure, and chemical reactivity of a

stable pentamethylcyclopentadienyl-substituted

phosphanylium ion: (pentamethylcyclopentadienyl) (tert-

butylamino)phosphanylium tetrachloroaluminate

AUTHOR(S):

Gudat, Dietrich; Nieger, Martin; Niecke, Edgar
Aporg, Chem. Inst., Univ. Bonn, Bonn, 5300/1, Fed

CORPORATE SOURCE:

Anorg. Chem. Inst., Univ. Bonn, Bonn, 5300/1, Fed.

Rep. Ger.

SOURCE:

Journal of the Chemical Society, Dalton Transactions: Inorganic Chemistry (1972-1999) (1989), (4), 693-700

CODEN: JCDTBI; ISSN: 0300-9246

DOCUMENT TYPE:

Journal

LANGUAGE:

English

II

OTHER SOURCE(S):

CASREACT 112:7649

GI

Stable phosphanylium salts of [P(NHCMe3)(C5Me5)]+ (I) were obtained via AB different routes, viz. Al2Cl6-promoted halide abstraction from a chlorophosphine precursor; displacement of chloride by the nucleofugic anion, OSO2CF3-; and protonation of an iminophosphine precursor. A crystalline product was isolated in case of the tetrachloroaluminate of I, and its structure was investigated by x-ray diffractometry. The results confirm the presence of discrete cations, featuring $\eta 2$ attachment of the C5Me5 ligand to P in the solid state. In solution, according to the results of NMR spectroscopic studies, the cation exhibits a fluxional structure with all 5 ring atoms becoming equivalent Investigations of the chemical reactivity of I include acid -base reactions and studies of the coordination chemical In addition to activity as both Lewis acid and base, which is a common feature for phosphanylium ions, I is the first two-coordinate P cation which reacted as a Broensted acid. Deprotonation initially gives the iminophosphine, P(:NCMe3)(C5Me5), which further reacts with I to yield a polycyclic cation, II, the structure of which was determined by x-ray diffraction. Reactions of I with transition metals involve oxidative addition of complex metal hydrides and coordination to reactive metal centers to give cationic complexes which are isolobal to transition metal carbene complexes. No evidence was obtained in these reactions to indicate any activation of the C5Me5-P bonds.

29-11 (Organometallic and Organometalloidal Compounds) Section cross-reference(s): 75

IT 104324-91-6P 123864-49-3P 123864-51-7P 123864-52-8P 123864-56-2P 123864-58-4P 123894-50-8P 123924-24-3P 123990-29-4P

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of)

IT 104324-91-6P

> RL: SPN (Synthetic preparation); PREP (Preparation) (preparation of)

104324-91-6 HCAPLUS RN

Phosphonamidous chloride, N-(1,1-dimethylethyl)-N-(1,2,3,4,5-pentamethyl-CN 2,4-cyclopentadien-1-yl)- (9CI) (CA INDEX NAME)

L51 ANSWER 14 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:

1988:570547 HCAPLUS Full-text

DOCUMENT NUMBER:

TITLE:

Associative and dissociative mechanisms for the reactions of N-tert-butyl-P-phenylphosphonamidic chloride with isopropylamine and tert-butylamine: competitive, kinetic, and stereochemical studies

AUTHOR(S):

CORPORATE SOURCE:

SOURCE:

Freeman, Sally; Harger, Martin J. P. Dep. Chem., Univ. Leicester, Leicester, LE1 7RH, UK Journal of the Chemical Society, Perkin Transactions

2: Physical Organic Chemistry (1988), (1), 81-90

CODEN: JCPKBH; ISSN: 0300-9580

PUBLISHER:

Royal Society of Chemistry

DOCUMENT TYPE: LANGUAGE:

Journal English

OTHER SOURCE(S):

CASREACT 109:170547

The substitution reactions of N-tert-butyl-P-phenylphosphonamidic chloride, AB PhP(O)(NHCMe3)Cl, with RNH2 (R = Me3C, Me2CH) can proceed by both associative and dissociative pathways. The associative pathway displays the characteristics expected of an SN2(P) mechanism, i.e. it is first-order in amine (nucleophile), it discriminates strongly against bulky amines (Me3CNH2), and it proceeds with complete stereospecificity. The dissociative pathway is less straightforward and embraces two mechanisms, both of which involve elimination-addition (EA). Both discriminate rather poorly between competing amines and form the substitution product nonstereospecifically, but they have different kinetic characteristics. One of the EA mechanisms is first-order in amine (base) and tends to be overshadowed by the SN2(P) reaction. With more hindered amines (Me3CNH2, EtMe2CNH2) however, steric hindrance makes the SN2(P) reaction less favorable and the EA mechanism becomes revealed more clearly proceeds with practically complete racemization. This is consistent with a simple EA mechanism in which the substitution product is derived from the free, sym. solvated, metaphosphonamidate intermediate. The other EA mechanism is second-order in amine (nucleophile and base) and is favored relative to the competing mechanisms by high concns. of amine. It involves preassocn. of the nucleophile with the conjugate base of the substrate and proceeds with extensive racemization.

CC 29-7 (Organometallic and Organometalloidal Compounds) Section cross-reference(s): 22

IT 116762-41-5P

RL: SPN (Synthetic preparation); PREP (Preparation) (preparation and salt formation of, with tert-butylamine or chiral methylbenzylamine)

IT 95980-86-2P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent) (preparation and sulfuration of)

IT 116762-44-8P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation, base hydrolysis, and ozonolysis of)

IT 2627-86-3

RL: PROC (Process)

(salt formation of, with phenylphosphonamidothioic acid derivative)

IT 95980-86-2P

RN 95980-86-2 HCAPLUS

CN Phosphonamidous chloride, N-(1,1-dimethylethyl)-P-phenyl- (9CI) (CA INDEX NAME)

C1 | Ph-P-NHBu-t DOCUMENT NUMBER:

CORPORATE SOURCE:

102:185163

TITLE:

Attempted synthesis of trimesitylphosphaethene; observations related to the mechanism of acid

catalyzed nucleophilic substitutions at

phosphorus (III)

AUTHOR(S):

Van der Knaap, Theodorus A.; Bickelhaupt, Friedrich Vakgroep Org. Chem., Vrije Univ., Amsterdam, 1081 HV,

SOURCE:

Phosphorus and Sulfur and the Related Elements (1984),

21(2), 227-36

CODEN: PREEDF; ISSN: 0308-664X

DOCUMENT TYPE:

Journal

LANGUAGE: English AΒ

Trimesitylphosphaethene (MesP:CMes2) (Mes = mesityl) is of interest as a sterically protected and presumably very stable phosphaalkene. Its synthesis was attempted along three different routes. The first two routes were modeled after the well-documented syntheses of phosphaalkenes by base catalyzed elimination of hydrogen chloride from MesPClCHMes2 (I). In the first approach, I could not be obtained from the precursor MesP(NEt2)CHMes2 by treatment with hydrogen chloride. Instead, the phosphonium salt [MesPH(NEt2)CHMes2]+Cl- (II) was formed; (II) is of interest as a "frozen" intermediate in the acid catalyzed nucleophilic substitution at phosphorus(III). The mechanistic implications of its formation and the reasons for its lack of reactivity are discussed. In the second approach, I was obtained from the reaction of MesPCl2 with α -potassiodimesitylmethane. However, several attempts to eliminate hydrogen chloride from I were unsuccessful. Similarly, the third route, aimed at the preparation of C1P: CMes2 from C12PCHMes2 (III) was thwarted because hydrogen chloride could not be eliminated from III. The unusual behavior of I, II and III can be explained by steric hindrance in these extremely crowded mols.

29-7 (Organometallic and Organometalloidal Compounds) CC

ST trimesitylphosphaethene attempted prepn; phosphoethene trimesityl attempted prepn; acid catalyzed nucleophilic substitution phosphorus; steric hindrance mesitylphosphene

IT 96156-61-5P

> RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation and reaction of, with hydrogen chloride)

733-07-3P 78204-84-9P 96156-60-4P 96156-64-8P ΙT 96156-65-9P 96156-67-1P 96156-68-2P 96156-69-3P 96156-70-6P RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of)

IT 96156-61-5P

> RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation and reaction of, with hydrogen chloride)

96156-61-5 HCAPLUS RN

Phosphinous amide, P-[bis(2,4,6-trimethylphenyl)methyl]-N,N-diethyl-P-CN (2,4,6-trimethylphenyl) - (9CI) (CA INDEX NAME)

IT 96156-60-4P 96156-69-3P 96156-70-6P

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of)

RN 96156-60-4 HCAPLUS

CN Phosphinous amide, P-[bis(2,4,6-trimethylphenyl)methyl]-N,N-diethyl-P-(2,4,6-trimethylphenyl)-, monohydrochloride (9CI) (CA INDEX NAME)

HCl

RN 96156-69-3 HCAPLUS

CN Pyrimido[1,2-a]azepinium, 1-[[bis(2,4,6-trimethylphenyl)methyl]chlorophosp hino]-2,3,4,6,7,8,9,10-octahydro-, chloride (9CI) (CA INDEX NAME)

● c1-

RN 96156-70-6 HCAPLUS

CN Phosphonamidous chloride, P-[bis(2,4,6-trimethylphenyl)methyl]-N,N-bis(trimethylsilyl)- (9CI) (CA INDEX NAME)

L51 ANSWER 16 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:

1980:620834 HCAPLUS Full-text

DOCUMENT NUMBER:

93:220834

TITLE:

Optically active trivalent phosphorus acid

esters: synthesis, chirality at phosphorus and some

transformations

AUTHOR(S):

Mikolajczyk, Marian

CORPORATE SOURCE:

Cent. Mol. Macromol. Stud., Pol. Acad. Sci., Lodz,

90-362, Pol.

Journal

SOURCE:

Pure and Applied Chemistry (1980), 52(4), 959-72

CODEN: PACHAS; ISSN: 0033-4545

DOCUMENT TYPE:

LANGUAGE: English

AB Optically active trivalent P acid esters [e.g., PhP(OMe)(Et)] and thio esters were prepared by 3 methods. These were asym. condensation of racemic trivalent P chlorides with achiral alcs. or thiols in the presence of chiral amines, asym. reaction of racemic chlorophosphines with menthol, and stereospecific preparation from optically active methylthioalkoxyphosphonium triflates. Optical purity and chirality at P were determined by chemical correlations. Nucleophilic substitution at chiral trivalent P occurs with inversion of configuration. Chiral tertiary phosphines (e.g., PhPMePr) of high optical purity were also prepared

CC 29-7 (Organometallic and Organometalloidal Compounds) Section cross-reference(s): 22

ST asym prepn phosphorus acid ester; thio ester phosphorus asym prepn; stereospecific prepn phosphorus acid ester; chirality phosphorus ester substitution

IT Asymmetric synthesis and induction

(of trivalent phosphorus acid esters and thio esters)

IT Stereochemistry

(stereospecificity, of preparation of trivalent phosphorus acid esters and thio esters)

IT 72315-67-4P 72315-69-6P 72315-73-2P 74171-25-8P 74184-50-2P 75466-61-4P 75466-62-5P 75466-63-6P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(preparation and reaction of, with base)

IT 52119-19-4P 72974-36-8P 74158-47-7P 75466-58-9P RL: RCT (Reactant); SPN (Synthetic preparation); PREP

(Preparation); RACT (Reactant or reagent)

(preparation and thionation of)

IT 1515-99-7P 17045-47-5P 21448-79-3P

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of, by alkylation of chiral phosphorus acid ester)

IT 41899-40-5P

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of, by elimination reaction of thioethyl phosphonium

salt)

IT 69460-42-0P

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of, by oxidation of phosphorus acid ester)

IT 55705-78-7P

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of, by thionation of phosphorus acid ester).

IT 6588-28-9 15849-83-9 15849-86-2

RL: RCT (Reactant); RACT (Reactant or reagent)

(reaction of, with alcs., in presence of chiral amines, chiral

phosphorus acid esters by)

IT 72974-36-8P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP

(Preparation); RACT (Reactant or reagent)

(preparation and thionation of)

RN 72974-36-8 HCAPLUS

CN Phosphinous amide, P-ethyl-N,N-dimethyl-P-phenyl-, (S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L51 ANSWER 17 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:

1977:164601 HCAPLUS Full-text

DOCUMENT NUMBER:

86:164601

TITLE:

Synthesis and characterization of dicoordinate

phosphorus cations. Compounds of the type

[(R2N)2P]+[Y]- and their congeners

AUTHOR(S):

Thomas, Michael G.; Schultz, Charles W.; Parry, R. W.

CORPORATE SOURCE:

Dep. Chem., Univ. Utah, Salt Lake City, UT, USA

Inorganic Chemistry (1977), '16(5), 994-1001

CODEN: INOCAJ; ISSN: 0020-1669

DOCUMENT TYPE:

Journal

LANGUAGE:

SOURCE:

English

On the basis of 1H and 31P NMR spectroscopy, IR data, measurements of elec. conductivity, and chemical information, the compound (Me2N)2PCl.AlCl3 is assigned the ionic structure [(Me2N)2P]+[AlCl4]-. The related compound Me2NPCl2.AlCl3 is assigned the structure [Me2NPCl]+[AlCl4]-. Salts of (Me2N)2P+ containing counterions such as PF6-, B2F7-, GaCl4-, and FeCl4- were prepared along with the GaCl4- salt of the Me2NPCl+ cation. The P in the cation Me2NPCl+ is the most deshielded P atom yet recorded. It has a chemical shift of -325 ppm from H3PO4. Both dicoordinate P cations are strong Lewis acids combining with a base such as (R2N)3P to give previously described cations such as [(R2N)3P-P(NR2)Y]+ where Y is NR2 or Cl. The dicoordinate

cations can also serve as ligands toward the metal atoms of metal carbonyls. Evidence for an N-P $p\pi$ - $p\pi$ bond is found with (R2N)2P+.

CC 78-7 (Inorganic Chemicals and Reactions)
Section cross-reference(s): 73

Section cross-

IT Lewis acids
 RL: RCT (Reactant); RACT (Reactant or reagent)

(phosphorus dicoordinate cations as)

IT 52653-69-7P 60594-82-3P 60594-84-5P 60594-92-5P 60607-14-9P

61770-32-9P 61770-33-0P 61770-34-1P 61770-35-2P

61770-36-3P 61788-02-1P 61788-03-2P 61788-05-4P

61788-06-5P

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of)

IT 61770-33-0P 61770-34-1P 61770-36-3P

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of)

RN 61770-33-0 HCAPLUS

CN Phosphinaminium, 1,1-bis(dimethylamino)-N,N,N-triethyl- (9CI) (CA INDEX

NAME)

Me2N-P-N+Et3

RN 61770-34-1 HCAPLUS

CN Pyridinium, 1-[bis(dimethylamino)phosphino]- (9CI) (CA INDEX NAME)

NMe2 P—NMe2

RN 61770-36-3 HCAPLUS

CN Phosphorodiamidous acid, tetramethyl-, ethyl ester, conjugate monoacid (9CI) (CA INDEX NAME)

NMe₂ Me₂N—P—OEt

● H+

L51 ANSWER 18 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN ACCESSION NUMBER: 1972:559645 HCAPLUS Full-text

DOCUMENT NUMBER:

77:159645

TITLE:

Lewis basicity of some difluorophosphines toward

borane

AUTHOR(S):

Foester, R.; Cohn, Kim

CORPORATE SOURCE:

Dep. Chem., Michigan State Univ., East Lansing, MI,

SOURCE:

Inorganic Chemistry (1972), 11(11), 2590-3

CODEN: INOCAJ; ISSN: 0020-1669

DOCUMENT TYPE:

LANGUAGE:

English

AΒ MeSPF2, (MeS) 2PF, MePF2 · BH3, MeSPF2 · BH3, and Me2PF · BH3 were prepared and characterized by 19F, 11B, 1H, and 31P NMR and ir spectroscopy as well as by stoichiometric data. Mass spectral data were also used to help characterize MeSPF2 and (MeS)2PF. A series of base displacement reactions established the base strengths toward borane as MePF2 > Me2NPF2 > MeOPF2 > MeSPF2 ≥ (MeS)2PF while 1JBP for the fluorophosphine-borane adducts decreases in the series Me2NPF2 > MeOPF2 > MePF2 > MeSPF2 > (MeS)2PF. The basicity of MePF2 is not mirrored by the value of the 1JBP coupling constant

CC 78-8 (Inorganic Chemicals and Reactions)

Lewis bases IT

RL: RCT (Reactant); RACT (Reactant or reagent)

(fluorophosphines as, with borane)

35512-81-3P 35512-89-1P 38627-26-8P 38627-27-9P IT 2851-73-2P

RL: SPN (Synthetic preparation); PREP (Preparation).

(preparation of)

IT 2851-73-2P

RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of)

2851-73-2 HCAPLUS RN

Boron, (dimethylphosphoramidous difluoride-N)trihydro-, (T-4)- (9CI) (CA CN INDEX NAME)

L51 ANSWER 19 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:

1967:28870 HCAPLUS Full-text 66:28870

DOCUMENT NUMBER: ORIGINAL REFERENCE NO.:

66:5511a,5514a

TITLE:

Reaction of insertion of a carbonyl group in

transition metal complexes by the action of a third

coordinating species: synthesis of

 π -cyclopentadienyltricarbonylacetylmolybdenum

or-tungsten derivatives

AUTHOR(S):

Capron-Cotigny, Ginette; Poilblanc, Rene

CORPORATE SOURCE: Fac. Sci., Toulouse, Fr.

SOURCE:

Comptes Rendus des Seances de l'Academie des Sciences, Serie C: Sciences Chimiques (1966), 263(15), 885-7

CODEN: CHDCAQ; ISSN: 0567-6541

DOCUMENT TYPE:

LANGUAGE:

Journal French

Lewis bases reacted readily $(20^\circ-60^\circ)$ and quant. with π -cyclopentadienyl carbonyls of Mo or W containing an entirely organic ligand, e.g. Me, in a manner termed insertion. π -C5H5WAc(CO)2PEt3, m. 42°, and IPEt3, m. 98°, IP[NMe2]3, m. 120°, and IP(OMe)3 where I is π -C5H5MoAc(CO)2- were prepared and their structures established by ir and N.M.R. studies.

CC 29 (Organometallic and Organometalloidal Compounds)

IT Bases, uses and miscellaneous

RL: USES (Uses)

(Lewis, carbonyl group insertion in transition metal complexes by rearrangement in presence of)

IT 554-70-1DP, Phosphine, triethyl-, complexes with molybdenum and tungsten 1608-26-0DP, Phosphorous triamide, hexamethyl-, molybdenum complexes 12110-00-8P

IT 1608-26-0DP, Phosphorous triamide, hexamethyl-, molybdenum complexes

RL: SPN (Synthetic preparation); PREP (Preparation)
 (preparation of)

RN 1608-26-0 HCAPLUS

CN Phosphorous triamide, N,N,N',N',N'',N''-hexamethyl- (CA INDEX NAME)

NMe₂ Me₂N—P—NMe₂

L51 ANSWER 20 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1965:401475 HCAPLUS Full-text

DOCUMENT NUMBER: 63:1475
ORIGINAL REFERENCE NO.: 63:231b-c

TITLE: Reactions giving zinc hydrogen ferrocyanide and its

method of preparation and ion exchange properties

AUTHOR(S): Tananaev, I. V.; Korol'kov, A. P.

CORPORATE SOURCE: M. V. Lomonosov Inst. Fine Chem. Technol., Moscow

SOURCE: Izvestiya Akademii Nauk SSSR, Neorganicheskie

Materialy (1965), 1(1), 100-7 CODEN: IVNMAW; ISSN: 0002-337X

DOCUMENT TYPE: Journal LANGUAGE: Russian

AB Solubility detns., potentiometric and conductometric titrns., and determination of the apparent vols. of precipitate show that the reaction of ZnSO4 with H4[Fe(CN)6] occurs in 2 steps, giving first Zn2[Fe(CN)6] and then H2Zn3[Fe(CN)6]2. In dilute solution the 2nd step is slow because an insol. film of product forms on the surface of the intermediate. With concentrated solns. both steps are rapid. In the presence of H2SO4 only the acid salt is formed. The precipitate peptizes on prolonged washing. The acid salt will

exchange H+ for Zn++ from solution CC 14 (Inorganic Chemicals and Reactions)

IT Base-exchanging substances or Cation-exchanging substances (zinc hexacyanoferrate(II) (Zn3H2[Fe(CN)6]2) as)

IT 19584-62-4P, Zinc hexacyanoferrate(II), Zn3H2[Fe(CN6]2 RL: PREP (Preparation)

(formation and base-exchanging properties of)

IT 2453-13-6P, Piperidine, 1,1'-(cyclohexylphosphinidene)di- 2453-13-6P, Phosphine, cyclohexyldipiperidino- 2453-16-9P, Phosphinous

chloride, cyclohexylpiperidino— 2453-17-0P, Piperidine,
1,1'-(dicyclohexyl-1,2-diphosphinediyl)di— 2453-19-2P, Phosphine
sulfide, cyclohexyldipiperidino— 2774-06-3P, Phosphonium,
cyclohexyldimethylpiperidino—, iodide— 13408-63-4P, Ferrate(II),
hexacyano— 92162-12-4P, Phosphorane, cyclohexyliododimethylpiperidino—
93815-30-6P, Phosphorane, cyclohexyliodomethyldipiperidino—
879646-69-2P, Phosphonium, cyclohexylmethyldipiperidino—, iodide
RL: PREP (Preparation)
(preparation of)

IT 2453-16-9P, Phosphinous chloride, cyclohexylpiperidino-RL: PREP (Preparation)

(preparation of)

RN 2453-16-9 HCAPLUS

CN Phosphinous chloride, cyclohexylpiperidino- (7CI, 8CI) (CA INDEX NAME)

L51 ANSWER 21 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1964:45364 HCAPLUS Full-text

DOCUMENT NUMBER: 60:45364
ORIGINAL REFERENCE NO.: 60:7915f-h

TITLE: Aliphatic 1,3-diamines

INVENTOR(S): Scott, Francis L.

PATENT ASSIGNEE(S): Pennsalt Chemicals Corp.

SOURCE: 3 pp.

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
	-			
US 3119872		19640128	US	19601005
PRIORITY APPLN. INFO.:			US	19601005

AB Aliphatic 1,3-diamines are prepared by catalytic hydrogenation of the condensation products of N2H4 with α,β-ethylenically unsatd. aldehydes or ketones. Aqueous alc. solns. of equimolar amts. of N2H4 and α,β-ethylenically unsatd. aldehydes or ketones at pH 6.0-8.0 are refluxed 0.5-10 hrs. Without isolation, the condensation products are hydrogenated at 50-100° and 100-300 lb./in.2 in the presence of a Raney Ni catalyst and a strong base as a cocatalyst (a base equivalent to, or stronger than NH4OH). In examples, 1,3-butanediamine was produced by condensing N2H4.H2O with crotonaldehyde, and hydrogenating the product with Raney Ni and NH3, and with Raney Ni and NaOH; 2-methyl-1,3- propanediamine was similarly produced from methacrolein; 1,3-pentanediamine from CH2:CHCOEt; 3-methyl-1,3-butanediamine from β-methylcrotonaldehyde; and 2-methyl-1,3-pentanediamine from 2-methyl-1-penten-3-one.

INCL 260583000

CC 33 (Aliphatic Compounds)

IT Bases

(catalysts from Raney Ni and strong, in hydrogenation of N2H4 reaction

```
products with \alpha, \beta-ethylenic aldehydes and ketones)
                                   2400-78-4P, 1,3-Propanediamine, 2-methyl-
IT
     589-37-7P, 1,3-Pentanediamine
     7319-05-3P, Phosphorous triamide, hexamethyl-, compound with borane
     15853-84-6P, Phosphorous triamide, hexaethyl-, compound with borane
     94485-30-0P, Pyrrolidine, 1,1',1''-phosphinidynetri-, compound with borane
     97437-22-4P, Piperidine, 1,1',1''-phosphinidynetri-, compound with borane
     101520-00-7P, Phosphorous triamide, hexabutyl-, compound with borane
     106847-15-8P, Phosphorous triamide, hexaphenyl-, compound with borane
     107014-58-4P, Phosphorous triamide, hexacyclohexyl-, compound with borane
     107065-13-4P, Phosphorous triamide, N,N',N''-trimethyl-N,N',N''-triphenyl-
     , compound with borane
                              108037-66-7P, Phosphorous triamide, hexabenzyl-,
     compound with borane 878792-56-4P, Borane, compound with
     hexaphenylphosphorous, triamide 878792-57-5P, Borane, compound
     with hexaethylphosphorous triamide 879631-34-2P, Borane, compound
     with 1,1',1''-phosphinidynetripiperidine 879631-48-8P, Borane,
     compound with hexacyclohexylphosphorous triamide 879631-55-7P,
     Borane, compound with hexabutylphosphorous triamide 879631-63-7P,
     Borane, compound with hexabenzylphosphorous triamide 879631-70-6P,
     Borane, compound with N,N',N''-trimethyl-N,N',N''-triphenylphosphorous
     triamide 879634-52-3P, Borane, compound with hexamethylphosphorous
     triamide
     RL: PREP (Preparation)
        (preparation of)
     878792-56-4P, Borane, compound with hexaphenylphosphorous, triamide
IT
     878792-57-5P, Borane, compound with hexaethylphosphorous triamide
     879631-34-2P, Borane, compound with 1,1',1''-
     phosphinidynetripiperidine 879631-48-8P, Borane, compound with
     hexacyclohexylphosphorous triamide 879631-55-7P, Borane, compound
     with hexabutylphosphorous triamide 879631-63-7P, Borane, compound
     with hexabenzylphosphorous triamide 879631-70-6P, Borane, compound
     with N,N',N''-trimethyl-N,N',N''-triphenylphosphorous triamide
     879634-52-3P, Borane, compound with hexamethylphosphorous triamide
     RL: PREP (Preparation)
        (preparation of)
RN
     878792-56-4 HCAPLUS
     Borane, compd. with hexaphenylphosphorous, triamide (7CI) (CA INDEX NAME)
CN
      NPh2
```

```
NPh2
Ph2N—P—NPh2
```

BH3

```
RN 878792-57-5 HCAPLUS
CN Borane, compd. with hexaethylphosphorous triamide (7CI) (CA INDEX NAME)
```

```
NEt2
|
Et2N—P—NEt2
```

BH3

RN 879631-34-2 HCAPLUS

CN Borane, compd. with 1,1',1''-phosphinidynetripiperidine (7CI) (CA INDEX NAME)

BH3

RN 879631-48-8 HCAPLUS

CN Borane, compd. with hexacyclohexylphosphorous triamide (7CI) (CA INDEX NAME)

● BH3

RN 879631-55-7 HCAPLUS

CN Borane, compd. with hexabutylphosphorous triamide (7CI) (CA INDEX NAME)

N (Bu-n) 2 (n-Bu) 2N—P—N (Bu-n) 2

BH:

RN 879631-63-7 HCAPLUS

CN Borane, compd. with hexabenzylphosphorous triamide (7CI) (CA INDEX NAME)

внз

RN 879631-70-6 HCAPLUS

Borane, compd. with N,N',N''-trimethyl-N,N',N''-triphenylphosphorous CN triamide (7CI) (CA INDEX NAME)

внз

879634-52-3 HCAPLUS RN

Borane, compd. with hexamethylphosphorous triamide (7CI) (CA INDEX NAME)

NMe2 Me2N—P—NMe2

внз

L51 ANSWER 22 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:

1964:9437 HCAPLUS Full-text

DOCUMENT NUMBER: 60:9437 ORIGINAL REFERENCE NO.: 60:1625c-f

TITLE: Action of tertiary nitrogen bases on several

phosphoric acid chlorides

Revel, Monique; Navech, Jacques; Vives, Jean Pierre AUTHOR(S):

CORPORATE SOURCE: Fac. Sci., Toulouse

SOURCE: Bulletin de la Societe Chimique de France (1963),

(10), 2327-31

CODEN: BSCFAS; ISSN: 0037-8968

DOCUMENT TYPE: Journal LANGUAGE: Unavailable

AΒ POC13 as well as alkyl and aryl phosphoryl dichlorides gave phosphorylammonium salts with tertiary amines. Their reaction with H2O and alcs. was studied; it appears that they behave towards alcs. as phosphorylating agents. The appropriate halophosphate (0.5 mole) in about 1 l. dry Et20 was treated dropwise with stirring with a large excess tertiary amine, the mixture kept 10 min. and filtered, and the residue recrystd. from MeCN to yield the following phosphorylammonium salts POCl3.3C5H5N (I), POCl3.Et3N,.POCl3.Me3N, POCl3 triquinoline (II), POCl3 trilutidine, PhOP(O)Cl2 (III).2C5H5N (IV), III.-2Me3N, III.2Et3N, ClCH2CH2OP(O)Cl2 (V).2C5H5N, V.2Me3N, V.2Et3N, EtOP(O)Cl2 (VI).2C5H5N, VI.2Me3N, VI.2Et3N, VI diquinoline, VI dilutidine, (BrCH2CH2O) (EtO) P(O) Cl. Me3N (in C6H6), (ClCH2CH2O) (PhO) P(O) Cl. Me3N (in C6H6). II dissolved in the min. amount H2O, diluted after several hrs. with 100 vols. H2O, and chromatographed two-dimensionally on paper demonstrated the formation of H3PO4 and quinoline-HCl. IV gave similarly PhOP(O)(OH)2 and [PhO(HO)P(O)]20. IV dissolved in excess MeOH and evaporated after several hrs. in vacuo gave an oily residue, which deposited a mixture of methyl phenyl phosphorylpyridinium chloride and pyridinium methomethyl- phenylphosphate. The oily mixture dissolved in N HCl, kept 2-3 hrs., and evaporated, and the residue extracted with Et2O gave oily C4H9O4P which in a little H2O with excess BaCO3 gave the Ba salt, C14H16O8P2Ba. IV dissolved in excess MeOH and evaporated and the oily residue dissolved in a small amount of H2O, diluted after several hrs. with 100 volume H2O, and chromatographed showed the presence of PhOP(O)(OH)2, MeOP(O)(OH)2, and C5H5N.HCl. I gave similarly with MeOH an oil, C2H7O4P, which yielded the Ba salt (C2H7OP)2Ba. II dissolved in MeOH, hydrolyzed, diluted with H2O, and chromatographed showed the presence of (MeO) 2P(O) (OH) and quinoline-HCl.

CC 35 (Noncondensed Aromatic Compounds)

IT Amines

(reactions of tertiary, with POCl3 or phosphorodichloridic acid esters)

IT 813-78-5P, Methyl phosphate, (MeO) 2 (HO) PO 4009-39-6P, Methyl phenyl 16368-97-1P, Phosphoric acid, phosphate, (MeO) (PhO) (HO) PO 17323-82-9P, Methyl barium phosphate, bis(2-ethylhexyl) Ph ester [(MeO)2PO2]2Ba 17323-82-9P, Barium methyl phosphate, Ba[O2P(OMe)2]2 91772-29-1P, Ammonium, trimethylphosphono, chloride, 2-bromoethyl Et ester 94628-65-6P, Ammonium, trimethylphosphono, chloride, 2-chloroethyl Ph 95725-60-3P, Pyridinium, 1,1'-phosphinicobis[- chloride], Et ester 95844-08-9P, 1-Methylpyridinium methyl phenyl phosphate 95875-35-7P, 1-Phosphonopyridinium chloride, methyl phenyl ester 96932-87-5P, Methyl barium phenyl phosphate, [(MeO)(PhO)PO2]2Ba 96932-87-5P, Barium methyl phenyl phosphate, Ba[O2P(OPh)(OMe)]2 97195-74-9P, Pyridinium, 1,1'-phosphinicobis[- chloride], Ph ester 97212-64-1P, Pyridinium, 1,1'-phosphinicobis[- chloride], 2-chloroethyl ester 98248-59-0P, Pyridinium, 1,1'-phosphinicobis[2,6-dimethyl- chloride], Et ester 740043-22-5P, Ammonium, phosphinicobis[trimethyl-, 2-chloroethyl ester 803648-46-6P, Ammonium, phosphinicobis[triethyl-, 2-chloroethyl ester 803650-97-7P, Ammonium, phosphinicobis[trimethyl-, Ph ester 804462-01-9P, Ammonium, phosphinicobis[triethyl-, Ph ester 805195-44-2P, Ammonium, phosphinicobis[triethyl-, Et ester 821008-52-0P, Pyridinium, 1,1',1''-phosphinylidynetris, [- chloride] 856584-14-0P, Ammonium, phosphinylidynetris[triethyl-, chloride RL: PREP (Preparation)

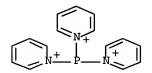
(preparation of)

IT 821008-52-0P, Pyridinium, 1,1',1''-phosphinylidynetris, [-chloride] 856584-14-0P, Ammonium, phosphinylidynetris[triethyl-,chloride

RL: PREP (Preparation) (preparation of)

RN 821008-52-0 HCAPLUS

Pyridinium, 1,1',1''-phosphinylidynetris, [- chloride] (7CI) CN NAME)



3 Cl-

RN 856584-14-0 HCAPLUS

CN Ammonium, phosphinylidynetris[triethyl-, chloride (7CI) (CA INDEX NAME)

N+Et3 Et3+N-P-N+Et3

L51 ANSWER 23 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:

1962:476076 HCAPLUS Full-text

DOCUMENT NUMBER:

57:76076

ORIGINAL REFERENCE NO.: 57:15145a-b

TITLE:

Preparation and reactions of some phosphobetaines

AUTHOR(S): CORPORATE SOURCE: Denney, Donald B.; Smith, Lois Chrisbacher

Rutgers Univ., New Brunswick, NJ

SOURCE:

Journal of Organic Chemistry (1962), 27, 3404-8

CODEN: JOCEAH; ISSN: 0022-3263

DOCUMENT TYPE:

LANGUAGE:

Journal Unavailable

Triphenylphosphine has been allowed to react with chloroacetic acid, β chloropropionic acid, and α -chlorobutyric acid. In each case the carboxyalkyltriphenylphosphonium salt was obtained. The salt from chloroacetic acid decarboxylated on heating or on treatment with base. The two other salts on treatment with base gave stable phosphobetaines. The chemistry of these materials is discussed. Triphenylphosphine and bromoacetic acid reacted, under several sets of conditions, to give triphenylphosphine oxide and acetyl bromide.

CC 33 (Organometallic and Organometalloidal Compounds)

IT 1031-15-8P, Phosphonium, methyltriphenyl, chloride 1636-14-2P, Phosphonous diamide, N,N,N',N'-tetraethyl-P-phenyl- 1636-15-3P, Phosphinous amide, N, N-diethyl-P, P-diphenyl- 2129-89-7P, Phosphine oxide, methyldiphenyl- 4073-31-8P, Phosphonamidous chloride, N,N-diethyl-P-phenyl- 4365-60-0P, Phosphonium, (2-carboxyethyl)triphenyl-, hydroxide, inner salt 6143-71-1P, Phosphonous diamide, N, N, N', N'-tetramethyl-P-phenyl- 7343-26-2P, Phosphonium, (carboxymethyl)triphenyl-, chloride 36626-29-6P, Phosphonium, (2-carboxyethyl)triphenyl-, chloride 60633-15-0P, Phosphonium,

(3-carboxypropyl)triphenyl-, hydroxide, inner salt

60633-18-3P, Phosphonium, (3-carboxypropyl)triphenyl-, chloride

88637-36-9P, Phosphonous diamide, N,N-diethyl-P-phenyl-N',N'-dipropyl-93137-76-9P, Phosphonous diamide, N,N,-diethyl-N',N'-dimethyl-P-phenyl-

94375-84-5P, Phosphonous diamide, P-phenyl-N,N,N',N'-tetrapropyl-

RL: PREP (Preparation)

(preparation of)

IT 1636-15-3P, Phosphinous amide, N,N-diethyl-P,P-diphenyl-

4073-31-8P, Phosphonamidous chloride, N,N-diethyl-P-phenyl-

RL: PREP (Preparation)

(preparation of)

RN 1636-15-3 HCAPLUS

CN Phosphinous amide, N, N-diethyl-P, P-diphenyl- (CA INDEX NAME)

Ph P— Ph Et—N— Et

RN 4073-31-8 HCAPLUS

CN Phosphonamidous chloride, N, N-diethyl-P-phenyl- (CA INDEX NAME)

Ph-P-NEt₂

L51 ANSWER 24 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:

1961:81609 HCAPLUS Full-text

DOCUMENT NUMBER:

55:81609

ORIGINAL REFERENCE NO.:

55:15431a-f

TITLE:

Reactions of naphthols and naphthylamines with

bisulfites (Bucherer reaction). V. Carbazole synthesis from naphthols or naphthylamines with phenylhydrazine

and bisulfites

AUTHOR(S):

Rieche, Alfred; Seeboth, Helmuth

SOURCE:

Ann. (1960), 638, 81-92

DOCUMENT TYPE:

Journal

LANGUAGE:

Unavailable

OTHER SOURCE(S):

CASREACT 55:81609

AB α-Naphthols or α-naphthylamines react with NaHSO3 and PhNHNH2 in aqueous solution to form 1-tetralone-3-sulfonic acid phenylhydrazones (XIV). These compds. are converted (influence of acids) partially to 1,2-benzocarbazole and partially to diamino compds. Bases cause conversion to phenylazonaphthalene or (also) diamino compds. Thus, 27 g. naphthionic acid is refluxed 6 h. with 11 g. PhNHNH2 and 200 g. 38% NaHSO3 solution, the product (after cooling) filtered, slurried in 200 mL. concentrated NaCl solution and filtered again. The crystals are dissolved in 350 mL. H2O and enough (AcO)2Ba solution added to precipitate all the sulfite and sulfate. The mixture is filtered and the filtrate treated with cation exchanger (Wofatit F) to convert the product to the free acid. Then 10% KHCO3 solution is added dropwise until the neutral point is reached. After acidification with a few ml. AcOH, the solution is

evaporated in vacuo. The yellow-brown residue is dissolved in 50 mL. H2O, the solution treated with C, filtered, and 100 mL. EtOH added to the filtrate to give 5.6 g. white needles of V. V (4.4 g.) is refluxed 20 min. with 100 mL. 20% KOH to give orange crystals, filtered off, and washed (cold H2O); the dry, pulverized compound (2.6 g.) is extracted twice with Et20; from the deep red Et20-extract is obtained (after evaporation) 1.3 g. 1-phenylazonaphthalene, red, m. 69.5° (EtOH). The Et2O-insol. portion is K 1-phenylazonaphthalene-4sulfonate, orange plates (EtOH). V (8 g.) is heated (steam bath) 6 h. with 100 mL. 30% HCl. The mixture turns deep-red at 1st, finally becoming yellow, and a grayish-white mass, white needles, and brown flakes precipitate The whole mixture is extracted with 100 mL. Et20. The dark Et20 solution is shaken with 40 mL. 2N NaOH and the extracted Et2O solution evaporated to dryness to give 0.6 g. 1,2-benzocarbazole, m. 225° (after sublimation). acid aqueous phase is filtered to give the insol. 1-amino-2-(4aminophenyl)naphthalene-4-sulfonic acid (XV), purified by slurrying with 25 mL. 96% EtOH, twice dissolving the insol. material with 2N NaOH, and precipitating with HCl, white needles, m. 280-2°. XV (via diazotization) gives the Na salt of 2-phenylnaphthalene-4-sulfonic acid (XVI)·2H2O, treated with concentrated HCl (16 h. at 140°) in a closed tube to give 2phenylnaphthalene. Treatment of β -naphthol or β -naphthylamine with NaHSO3 and PhNHNH2 in H2O gives 3,4-benzocarbazole; 1,2-dihydro-3,4-benzocarbazole-2sulfonic acid forms as an intermediate. This carbazole synthesis was found to proceed analogously to the indole synthesis of Emil Fischer.

CC 10F (Organic Chemistry: Condensed Carbocyclic Compounds)

1,2-Naphthalenedisulfonic acid, 1,2,3,4-tetrahydro-4-oxo-, phenylhydrazone, di-K salt
RL: PREP (Preparation)

205-25-4P, 7H-Benzo[c]carbazole 239-01-0P, 11H-Benzo[a] carbazole ΙT 2653-70-5P, 1-Naphthaleneazobenzene 92967-07-2P, 2-Naphthalenesulfonic acid, 1,2,3,4-tetrahydro-4-oxo-, phenylhydrazone 114380-67-5P, Naphthionic acid, 3-(p-aminophenyl)-114380-68-6P, Pyridine, compound with 3-(p-aminophenyl)naphthionic acid 114380-68-6P, Naphthionic acid, 3-(p-aminophenyl)-, compound with pyridine 116568-54-8P, 1-Naphthalenesulfonic acid, 3-phenyl-, sodium salt 856639-35-5P, Hydrazine, phenyl-, compound with 5,6-dihydro-7P-benzo[c]carbazole-5-sulfonic acid 857220-60-1P, 1-Naphthalenesulfonic acid, 4-phenylazo-, potassium salt RL: PREP (Preparation)

(preparation of)

IT 112486-22-3, 7H-Benzo[c]carbazole-5-sulfonic acid, 5,6-dihydro-(salts)

856639-35-5P, Hydrazine, phenyl-, compound with 5,6-dihydro-7Pbenzo[c]carbazole-5-sulfonic acid
RL: PREP (Preparation)

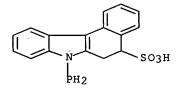
(preparation of)

RN 856639-35-5 HCAPLUS

CN Hydrazine, phenyl-, compd. with 5,6-dihydro-7P-benzo[c]carbazole-5-sulfonic acid (6CI) (CA INDEX NAME)

CM 1

CRN 856639-34-4 CMF C16 H14 N O3 P S



CM

100-63-0 CRN C6 H8 N2 CMF

H2N-NH-Ph

L51 ANSWER 25 OF 25 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1958:97992 HCAPLUS Full-text

DOCUMENT NUMBER: 52:97992

52:17275i,17276a-i,17277a-b

ORIGINAL REFERENCE NO.:

TITLE:

Phosphoramidic halides. Phosphorylating agents derived

from morpholine

AUTHOR(S):

Montgomery, H. A. C.; Turnbull, J. H.

CORPORATE SOURCE:

Univ. Birmingham, UK

SOURCE:

Journal of the Chemical Society (1958) 1963-7

CODEN: JCSOA9; ISSN: 0368-1769

DOCUMENT TYPE:

LANGUAGE:

Journal Unavailable

OTHER SOURCE(S):

CASREACT 52:97992 cf. C.A. 51, 16488i. Mild acid hydrolysis of phosphorodimorpholidates (I), (C4H8NO)2PO2R, gave the corresponding dihydrogen phosphates (II), RH2PO4, conveniently isolated as their cyclohexylammonium (IIa) or phenylacetamidinium (IIb) salts. A sterically hindered tertiary base was used throughout to avoid dealkylation. Phosphoramidic halides (III), (PhNR)2POX, derived from aromatic amines appeared to have limited value as phosphorylating agents. POBr3 was prepared by the method of Gerrard, et al. (C.A. 41, 7292f), and 2,6-lutidine (IV) was purified according to Biddiscombe, et al. (C.A. 49, 5465h). Morpholine (35 g.) added gradually to 29 g. POBr3 in 120 ml. CHCl3 at 10° and the mixture stirred 4 hrs. at room temperature, the filtered solution evaporated to 50 ml. and kept at 0° (dry atmospheric) gave a suitable phosphorylation preparation of phosphorodimorpholidic bromide (V). H2C:CHCH2OH (3 ml.) in 4 ml. IV kept 5 hrs. with 17.4 ml. V (from 6.4 g. POBr3) and the volatile components evaporated in vacuo, the residue diluted with 40 ml. Et20, and filtered gave 2.6 g. I (R = H2C:CHCH2), b0.002 113-17°, n17D 1.4930. Similarly were prepared I (R = Et) (IVa), $b0.003 \ 102-8^{\circ}$, m. 48° and I (R = PhCH2CH2), n18.5D 1.5231 (chromatographed on silica gel and eluted with alc.). Cyclohexanol phosphorylated in the presence of (PhCH2)3N, the product chromatographed on silica gel, and crystallized (C6H6-petr. ether) gave I (R = cyclohexyl) (IVb), m. 53°. Similarly was prepared I (R = Me2C:CHCH2) as a sirup, C13H25N2O4P. IV (6 ml.) and 15 ml. V (from 4.5 g. POBr3) in 15 ml. CHCl3 warmed 15 min. at 40° with 2.0 g. 2-(4-methyl-5-thiazolyl)ethanol HCl salt (cf. Williams, et al., C.A. 29, 33819) and the mixture treated with 0.3

ml. H2O and excess petr. ether, the filtered solution evaporated, and the residue chromatographed in C6H6 over silica gel gave 1.5 g. I [R = 2-(4methyl-5-thiazolyl)ethyl], C14H24N3O4PS; dipicrolonate, m. 181° (alc.-Et2O). Morpholine (71 q.) added slowly to 19 ml. POCl3 in 200 ml. C6H6 at 10-20° and the mixture stirred 3 hrs., the filtered solution evaporated, and the residue distilled in 10 g. portions gave 32 g. phosphorodimorpholidic chloride (VI), b0.02 137-40° m. 81° (cyclohexane). EtOH (7 ml.), 4.5 ml. IV, and 8.5 g. VI refluxed 16 hrs. and the mixture evaporated, the residue extracted with Et20, and the product distilled gave 4 g. Ia. Cholesterol (5.4 g.), 10 ml. C5H5N, and 3.5 g. VI heated 17 hrs. at 76° (CCl4 bath) and the product diluted with H2O, the mixture filtered, and the precipitate crystallized (petr. ether) gave 3.7 g. I (R = cholesteryl) (VIa), m. 153°. Treatment of 1 mole Cl2PO2Ph with 4 moles morpholine at 20-30° and crystallization (cyclohexane) of the product gave I (R = Ph) (VIb), m. 84° (cf. Audrieth and Toy, C.A. 36, 44326). Similarly, 100 mg. cholesteryl phosphorodichloridate (cf. M., et al., C.A. 51, 6668h) warmed 1 hr. with 0.07 ml. morpholine in 0.8 ml. C6H6 and the filtered solution evaporated gave VIa. EtOH (23 g.) stirred 30 min. in 153 g. POCl3 at 0° and the product distilled gave 41 g. Cl2PO2Et (VII), b12 62-5°. VII (13 g.) in 200 ml. Et20 at $10-15^{\circ}$ treated with 27 g. morpholine and the filtered solution evaporated gave 7.2 g. IVa. Cyclohexanol (10 g.) and 11 g. IV in 20 ml. CCl4 kept 1 hr. at $0-10^{\circ}$ with 15 g. POCl3 in 100 ml. CCl4 and the filtered solution evaporated gave 20 g. cyclohexylphosphorodichloridate (VIII), decomposed on vacuum distillation to cyclohexene. VIII (10 g.) in 160 ml. CCl4 treated with 17 g. morpholine at $0-10^{\circ}$ and the mixture stirred 2 hrs. at room temperature, the filtered solution evaporated, and the product crystallized (C6H6-petr. ether) yielded 10 g. hygroscopic IVb. I (500 mg.) in 5 ml. H2O percolated in 1-2 hrs. through Amberlite IR-120 resin (H+ form) at 60° and the filtrates evaporated gave II [R, m.p. (solvent), IIa (IIb) and m.p. (solvent) given]: Et, sirup, 2 C6H13N, 188° (dilute Me2CO) [2 C8H10N, 157° (alc. Et20)]; Ph, 94° (CHCl3), 2 C6H13N, 211°; cyclohexyl, 86° (C6H6-C6H12), 2 C6H13N, 212° (alc.); H2C:CHCH2, sirup, 2 C6H13N, 175° (decomposition) (dilute Me2CO); PhCH2CH2, sirup, C6H13N, 177° (dilute Me2CO). VIb (530 mg.) in 5 ml. H2O treated 30 min. with Amberlite IR-120 resin (H+ form) and the solution evaporated yielded 290 mg. C4H8NOPHO2R; cyclohexylammonium salt, C10H14NO4P.C6H13N, m. 202° (dilute Me2CO). Cyclohexylphosphorodichloridate (10 g.) in 50 ml. CCl4 treated 30 min. with 8.5 g. tert-BuOH and 1.0 ml. $\rm H2O$ at $\rm 50^{\circ}$ and the solvent evaporated, the oily residue taken up in a slight excess of saturated aqueous NaHCO3 and the solution filtered through Amberlite, evaporated, and the residue crystallized (CHCl3C6H12) gave 4.8 g. H2P(C6H11)O4, m. 85°. C6H11OH (20 g.) and 21 g. IV in 40 ml. CCl4 gradually added at 0° to 15 g. POCl3 and 2 g. IV in 200 ml. CC14 and the mixture kept at room temperature overnight, the filtered solution shaken at 0° with M KHSO4 and the dried (Na2SO4) solution evaporated, the residue heated 30 min. at 90° with 10.5 g. tert-BuOH and the solution evaporated in vacuo at room temperature, the residue extracted with 80 ml. 2.5N NaOH, and the extract acidified with AcOH and treated with C6H11NH2 gave 4.7 g. cyclohexylammonium dicyclohexyl phosphate, C12H23O4P.C6H13N, m. 211° (EtOH-Et2O). Solvolysis of 180 mg. VIa by refluxing 50 hrs. in AcOH and diluting of the product with H2O gave 105 mg. 3β -acetoxy-5-cholestene (M., et al., loc. cit.), also obtained by heating VIa 20 min. at 100° with 90% HCO2H or 15 min. at 90° with 2N HCl in 80% AcOH. PhNHMe (214 g.) refluxed 1 hr. in 220 ml. PhMe with 77 g. POCl3 and the cooled, filtered solution evaporated and distilled yielded 90 g. III (R = Me, X = Cl) (IX), b0.03 149-51°, n20D 1.5851. IV (4.5 ml.) and 9.8 g. IX refluxed 16 hrs. in 8 ml. alc. and the mixture evaporated, the residue extracted with Et20 and the product distilled yielded 5.3 g. III (R = Me, X = OEt) (X), b0.03 145-8°, n18D 1.5631. X was recovered unchanged after prolonged treatment with Amberlite suggesting that delocalization of the lone-pair electrons on N by aromatic resonance protects the P-N bond from proton attack.

10G (Organic Chemistry: Heterocyclic Compounds)

CC

5-Thiazoleethanol, 4-methyl-, dipicrolonate IT Phosphonic acid, morpholino-, cyclohexylamine salt RL: PREP (Preparation) 856792-24-0, 2817-45-0, Phosphoramidic acid IT 1,5-Benzothiazepine, 2,3-dihydro-(derivs.) 7664-38-2, Phosphoric acid 13779-49-2, Phosphorodichloridic IT acid (esters, and other derivs.) 6913-01-5, Phosphinic acid, dimorpholino-IT (esters, hydrolysis of) 701-64-4P, Phenyl phosphate, (PhO)(HO)2PO 1498-51-7P, Ethyl IT phosphorodichloridate 3694-53-9P, Phosphorodiamidic acid, N,N'-dimethyl-N,N'-diphenyl-, ethyl ester 6787-44-6P, Phosphinic bromide, dimorpholino- 6901-51-5P, Cholesteryl phosphorodichloridate 7264-91-7P, Allyl 7264-90-6P, Phosphinic chloride, dimorpholinoalcohol, dimorpholinophosphinate 7264-92-8P, Allyl phosphate, 18110-43-5P, (C3H5O) (HO) 2PO, bis (cyclohexylamine) salt Phenethyl phosphate, (C8H9O) (HO) 2PO 25022-72-4P, Allyl phosphate, 46731-55-9P, Phosphonic acid, morpholino-, (C3H5O) (HO) 2PO 57775-14-1P, Phenyl phosphate, (PhO) (HO) 2PO, compds. with phenyl ester 58245-46-8P, Phosphorodiamidic chloride, cyclohexylamine N, N'-dimethyl-N, N'-diphenyl-86240-42-8P, Cyclohexyl 109446-70-0P, Phenethyl phosphate, cyclohexylamine phosphorodichloridate 112688-81-0P, 2-Buten-1-ol, 3-methyl-, 113977-26-7P, Cyclohexanol, dimorpholinophosphinate 114794-67-1P, Phenethyl alcohol, dimorpholinophosphinate dimorpholinophosphinate 860175-22-0P, 5-Thiazoleethanol, 4-methyl-, dimorpholinophosphinate 860226-48-8P, Picrolonic acid , compound with 2-(4-methyl-5-thiazolyl)ethyl dimorpholinophosphinate RL: PREP (Preparation) (preparation of) 1623-22-9P, Cyclohexyl phosphate IT RL: PREP (Preparation) (preparation of (C6H110)2(HO)PO and (C6H110)(HO)2PO and their cyclohexylamine salts) 860175-22-0P, 5-Thiazoleethanol, 4-methyl-, IT dimorpholinophosphinate RL: PREP (Preparation) (preparation of) 860175-22-0 HCAPLUS RN5-Thiazoleethanol, 4-methyl-, dimorpholinophosphinate (6CI) (CA INDEX CNNAME)

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(FILE 'HOME' ENTERED AT 11:09:27 ON 05 OCT 2007)

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L3 23 SEA ABB=ON PLU=ON L2 AND P/ELS

D SCA

L4 1580012 SEA ABB=ON PLU=ON P/ELS

L5 STR

L9

L*** DEL STR L5

L6 50 SEA SUB=L4 SSS SAM L5

L7 0 SEA ABB=ON PLU=ON L6 AND L3

FILE 'MARPAT' ENTERED AT 11:14:56 ON 05 OCT 2007

FILE 'CASREACT' ENTERED AT 11:15:21 ON 05 OCT 2007 E US2004-500145/AP,PRN

FILE 'CAPLUS' ENTERED AT 11:43:37 ON 05 OCT 2007 E PHOSPHINES/CT E E3+ALL

FILE 'HCAPLUS' ENTERED AT 11:44:02 ON 05 OCT 2007 68779 SEA ABB=ON PLU=ON PHOSPHINES+PFT,NT/CT

L10 1 SEA ABB=ON PLU=ON L1 AND L9

D KWIC

L11 7867 SEA ABB=ON PLU=ON PHOSPHINES+PFT, NT/CT(L) PREP+NT/RL

L12 0 SEA ABB=ON PLU=ON L11 AND L1

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E PHOSPHORUS ESTER DIAMIDE/CT E AMINOPHOSPHINES/CT
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O SEA ABB=ON PLU=ON PHOSPHORUS ESTER DIAMIDE
L13
    FILE 'REGISTRY' ENTERED AT 11:52:13 ON 05 OCT 2007
             1 SEA ABB=ON PLU=ON ?PHOSPHORUS ESTER?
L14
               D SCA
           446 SEA ABB=ON PLU=ON ?AMINOPHOSPHIN?/CNS
             O SEA ABB=ON PLU=ON ?AMINOPHOSPHINE CHLORIDE/CNS
L16
            33 SEA ABB=ON PLU=ON ?AMINOPHOSPHINE/CNS
L17
           0 SEA ABB=ON PLU=ON PHOSPHORUS ESTER/CNS
L18
            0 SEA ABB=ON PLU=ON PHOSPHORUSESTER/CNS
L19
L20
            6 SEA ABB=ON PLU=ON PHOSPHORUS (1W) ESTER/CNS
            O SEA ABB=ON PLU=ON PHOSPHOESTER/CNS
L21
L22
               STR
           50 SEA SUB=L4 SSS SAM L22
L23
         6867 SEA SUB=L4 SSS FUL L22
L24
    FILE 'CAPLUS' ENTERED AT 12:37:58 ON 05 OCT 2007
          2789 SEA ABB=ON PLU=ON L24(L)PREP+NT/RL
L25
             1 SEA ABB=ON PLU=ON L25 AND L1
L26
               D HITSTR
    FILE 'HCAPLUS' ENTERED AT 12:40:26 ON 05 OCT 2007
        378500 SEA ABB=ON PLU=ON ACIDS+PFT,NT1/CT
L27 .
          4202 SEA ABB=ON PLU=ON ACIDS+PFT, NT1/CT(L) REM/RL
L29 .
         34370 SEA ABB=ON PLU=ON ACIDS+PFT, NT1/CT(L)PREP+NT/RL
L30
          194 SEA ABB=ON PLU=ON L28 AND L29
             0 SEA ABB=ON PLU=ON L30 AND L1
L31
            1 SEA ABB=ON PLU=ON L25 AND L1
L32
L33
            1 SEA ABB=ON PLU=ON L27 AND L1
L34
            O SEA ABB=ON PLU=ON L28 AND L1
       0 SEA ABB=ON PLU=ON L28 OR L29
38378 SEA ABB=ON PLU=ON L28 AND L1
L35
L36 38
               E BASES+ALL/CT
       22956 SEA ABB=ON PLU=ON BASES+PFT,NT/CT
L38
L39
           1 SEA ABB=ON PLU=ON L38 AND L1
L40
             9 SEA ABB=ON PLU=ON L25 AND L38
L41
            65 SEA ABB=ON PLU=ON L27 AND L25
             1 SEA ABB=ON PLU=ON L41 AND L38
L42
             1 SEA ABB=ON PLU=ON L42 AND L1
L43
               E IONIC LIQUIDS/CT
               E E3+ALL
          5873 SEA ABB=ON PLU=ON IONIC LIQUIDS+PFT,NT/CT
               E IONIC FLUIDS/CT
             3 SEA ABB=ON PLU=ON L25 AND L44
L45
            0 SEA ABB=ON PLU=ON L45 AND L1
5 SEA ABB=ON PLU=ON L25 AND (L44 OR IONIC(2A)(LIQUID OR FLUID)
L46
L47
             OR (LIQUID OR MOLTEN) (2A) SALT)
L48
            1 SEA ABB=ON PLU=ON L47 AND L1
L49
            13 SEA ABB=ON PLU=ON L47 OR L40
L50
            14 SEA ABB=ON PLU=ON L25 AND (L27 OR ACID) AND (L38 OR BASE)
               AND SALT
L51
            25 SEA ABB=ON PLU=ON L49 OR L50
     FILE 'HCAPLUS' ENTERED AT 12:49:01 ON 05 OCT 2007
               D QUE L51
               D L51 IBIB ABS HITIND HITSTR TOT
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